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THE USE OF AOTF-NIR SPECTROMETERS TO ANALYZE FUELS

PHASE I. INSTRUMENT SELECTION AND PRELIMINARY CALIBRATIONS

INTERIM REPORT
TFLRF No. 313

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13. ABSTRACT (Maximum 200 words)

The U.S. Army has a need for analytical instrumentation that can assess the quality of fuels and lubricants both in the field and in near-the-battlefield conditions. Near-infrared (NIR) spectroscopy was identified as one analytical technique with the potential to meet the Army's requirements. The Army initiated a program to rigorously evaluate the feasibility of using NIR in the analysis of diesel fuels. For this program, the Army specified the use of acousto-optic tunable filter (AOTF)-based NIR instruments. Fuel samples totaling 427 were collected and analyzed for several common fuel properties. Three AOTF-NIR spectrometers were evaluated, and an additional six instruments were purchased based on the initial evaluation. This report presents the results of the fuel analyses and the instrument evaluations.

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EXECUTIVE SUMMARY

Problem: The evaluation of fuel quality in the field has been a continuous problem, even in recent military operations in Grenada and Panama. The 1991 operations Desert Shield and Desert Storm emphasized the necessity of identifying a method to evaluate specific fuel properties both rapidly and accurately.

Objective: The objective of this project is to evaluate and demonstrate the usefulness of AOTF-NIR spectroscopy to measure several common properties of diesel fuel and kerosene.

Importance of Project: The capability to rapidly determine several important engine fuel properties can reduce the frequency and severity of fuel-related equipment failures. It will also increase the confidence of the user when using undocumented fuel sources.

Technical Approach: AOTF-NIR spectrometers were purchased and comparatively evaluated using previously analyzed fuel samples. Instrument calibration and validation calculations were made and used to evaluate the performance of each instrument.

Accomplishments: Fuel samples totaling 427 were collected and analyzed for several common fuel properties. AOTF-NIR spectrometers were purchased from three instrument manufacturers. The three instruments were comparatively evaluated to determine the source for purchase of additional spectrometers. Based on the preliminary evaluation results, six additional spectrometers were purchased: three instruments were purchased from each of two of the original instrument manufacturers.

Military Impact: The results of this phase of the project provided both a demonstration of the capability of NIR to measure the desired fuel properties and a basis for selection of the sources of additional instrument purchases, thus assuring that the additional instruments will meet Army requirements.

FOREWORD/ACKNOWLEDGMENTS

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I. INTRODUCTION AND BACKGROUND

Military operations in Grenada and Panama in the 1980's and the 1991 Operations Desert Shield and Desert Storm (ODS) all suffered from the lack of proper fuel quality evaluation in the field, especially with regard to fuels provided by host nations.

Based on this experience, the U.S. Army expressed the urgency in identifying equipment that could assess the quality of fuels and lubricants both in the field and in near-battlefield conditions.(1-4)* Since that time, the Army has been working to develop a highly mobile test capability.(5) A recent project described a best technical approach (BTA) to meet the Army's proposed mobile petroleum testing requirement, known as the Petroleum Quality Analysis System (PQAS).(6) According to this BTA, the testing equipment should perform relatively quick and simple analyses and preferably be commercially available. The key guidelines for the PQAS may be summarized as follows:

- Emphasis is placed on the evaluation of fuels used in Army aviation and ground vehicles and support equipment.
- While primary interest is placed on the evaluation of fuels, the determination of lubricant suitability for specific use is also important.
- Both quick, on-the-spot determinations and in-depth analytical capabilities should be assessed.
- Procedures and technology should be developed where needed.

Near-infrared (NIR) spectroscopy was identified as one analytical technique with the potential to meet the requirements of the PQAS. Several characteristics of NIR make it useful for this application. These include the use of quartz optics and fiber optics, which make it less sensitive

* Underscored numbers in parentheses refer to the list of references at the end of this report.

to environmental conditions and more readily adaptable to field use; and high energy light sources and low noise detectors for a relatively high signal-to-noise ratio. State-of-the-art microcomputers, together with new computer software to perform multivariate statistical analysis/chemometrics, make it possible to quantitate these small differences in spectra, i.e., differences in the chemical composition and structure of the samples.

Figure 1 is the NIR spectra of four diesel fuels. As shown in this figure, the peaks in a near-infrared spectrum are typically broad and often overlapping. Historically, this characteristic of NIR spectra has kept them from yielding significant amounts of useful information. However, the NIR spectroscopic region is attractive for the analysis of hydrocarbon fuels since most of the absorption bands in this region are the result of overtones or combination bands of carbon-hydrogen (C-H) stretching vibrations. In addition, the absorptivity of these bands is largely independent of the remainder of the molecule but is dependent upon the concentration of the absorbing functionality.(7) Despite the relatively poor peak resolution, the vibrations of C-H

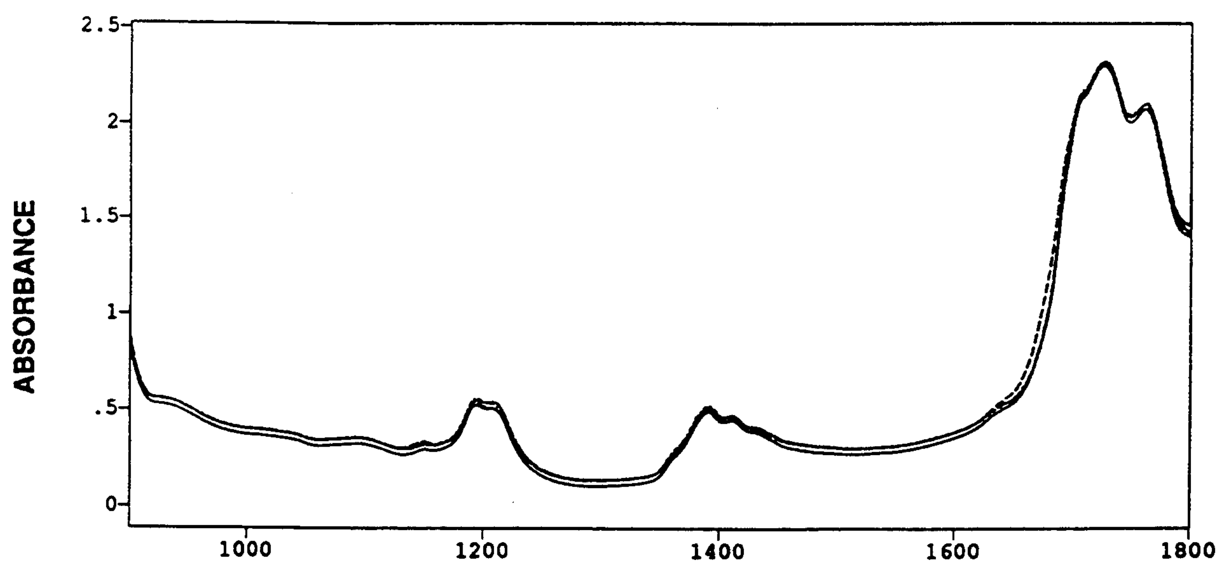

Figure 1. <u>**NIR spectra of four diesel fuels (transmittance mode, 900 nm to 1,800 nm)**</u>

bonds in different functional groups and different molecular environments will yield distinct transitions. Each of these transitions will make an accompanying contribution to the total spectrum for the sample. If the sample contains C-H, nitrogen-hydrogen (N-H), or oxygen-hydrogen (O-H) bonds and the analyte concentration is greater than about 0.1 percent of the total, then NIR will very likely be a useful analytical method for that analyte. In those instances where a property is being measured (such as cetane number) and not a specific component (such as aromatic hydrocarbons), a relationship must exist between the bulk chemical structure of the sample and the property being measured in order for NIR to be used in the measurement of that property. For example, in the relationship between chemical structure of a gasoline and the octane rating of the gasoline, higher aromatic and branched chain hydrocarbon content will increase the octane rating, while higher straight chain content will lower the octane rating. The opposite relationship is true for diesel fuels and cetane rating.

Much of the earliest work with NIR was in the field of agriculture in applications such as the measurement of the amount of protein in wheat.(8-10) Recently, NIR was investigated as a tool for analyzing gasoline. Typical uses for NIR in the analysis of gasoline include the measurement of octane number, aromatics content, density, vapor pressure, distillation, and oxygenate content.(11-17) In a similar study, Lysaght et al (18) evaluated the use of NIR in measuring the saturate and aromatic content and the freezing point of jet fuels. To date, little work has been reported on the use of NIR to measure the properties of diesel fuel and/or kerosene type fuels used for ground vehicle propulsion.

A preliminary study was conducted by the Army to evaluate the usefulness of NIR in measuring various properties of diesel fuel.(19) In this study, calibration results varied from poor to excellent. Flash point was not successfully modeled whereas density had a correlation coefficient of 0.99. Some properties such as cetane number and aromatics content achieved acceptable correlations that were lower than those for the other properties. Much of the reduced accuracy of these calibrations was attributed to the imprecision of the standard laboratory test methods utilized in measuring these properties. As the primary calibration data are improved, the correlations using NIR will also improve. Calibration models for each property were developed

and entered into the NIR spectrometer. Using these models, it was shown that a single NIR analysis could estimate values for several fuel properties at one time.

Based on the success of the preliminary study, the Army initiated a program to more rigorously evaluate the feasibility of using NIR spectroscopy in the analysis of diesel fuels. The funding for this project was specified in the U.S. House of Representatives, Department of Defense Appropriations Bill, 1993, Report of the Committee of Appropriations to accompany H.R. 5504, June 29, 1992. The appropriation included the following wording:

> "To assure the quality of fuel supplies, the Army employs extensive laboratory and field test facilities...New technology has been developed that automates many of the laboratory tests and significantly reduces investment for equipment and manpower. Acousto-Optic Tunable Filter (AOTF) technology achieves a consolidated multipurpose test capability in a single highly portable unit."

For this program, the Army specified the use of acousto-optic tunable filter (AOTF)-based NIR instruments. The AOTF is a compact, solid-state monochromator. It consists of a piezo electric transducer bonded to a propagating crystal. Sound waves are produced in the propagating crystal by applying a radio frequency drive to the transducer. Periodic pressure variations produced by the acoustic wave create corresponding variations in the refractive index of the crystal. The crystal then acts like a thick diffraction grating: light of a specific wavelength is bent and rotated in polarization. Since the line spacing is a function of frequency, it is possible to tune the optical frequency by tuning the input frequency. AOTF-NIR offers several advantages over non-AOTF instruments. These include the absence of moving parts, lower maintenance costs, greater spectral resolution, reduced size, and increased ruggedness. A more detailed discussion of AOTF technology can be found in References 20 through 23.

II. APPROACH

The initial phase of the project entailed the writing of a purchase specification, statement of work, and a request for quotation (RFQ) for the purchase of AOTF-NIR instruments for evaluation. The RFQ responses were studied and those instruments which met the Army's needs were identified. An evaluation instrument was purchased from each of three instrument suppliers. These instruments were evaluated head-to-head using a selected set of fuel samples. Based on these results, six additional instruments were purchased for more detailed evaluation, calibration transfer studies, and field tests.

For this phase of the project, 427 middle distillate fuel samples were collected from a wide range of sources. This resulted in a large and diverse set of test fuels. Each fuel was tested according to a predetermined protocol. Preliminary calibration models were developed with a selected subset of the test fuels. The preliminary calibration models were used for the comparative evaluation of the first three instruments prior to the purchase of additional instruments.

III. INSTRUMENT SELECTION

For the first round of instrument purchases, RFQ's were sent to eight instrument manufacturers. Responses were received from five companies; however, only three of the five responders offered to supply an AOTF-NIR. One instrument was purchased from each of those three instrument manufacturers. TABLE 1 contains a description of the three instruments purchased.

TABLE 1. AOTF-NIR Instruments

Manufacturer and Model	Spectral Range	Additional Information
Bran & Luebbe, InfraPrime Lab	900 to 1,700 nanometers	Dual beam instrument. Uses cuvettes. 1-cm path length.
Brimrose, Luminar 2000	900 to 1,600 nanometers	Transreflectance. Fiber optic probe. 1-cm path length.
Infrared Fiber Systems, Prizma 2600 M	900 to 2,600 nanometers	Transreflectance. Fiber optic probe. 1-cm path length.

IV. PRELIMINARY CALIBRATIONS

For this phase of the project, 427 fuel samples were obtained and analyzed according to the list of properties in TABLE 2: 214 samples were used to calibrate the instruments, and the remaining 213 samples were used for validation of the instrument calibration models. TABLE 3 lists descriptive statistics for these test fuels. The test fuels were first ranked by density. Every other fuel was then assigned to the validation set. This procedure assured a wide range of properties in both the calibration and validation sets. (See Reference 24 for a description and discussion of the fuels used in this project.)

Calibration models were developed on each instrument and then used to estimate the properties of the validation fuels. All of the chemometric calculations (calibrations and validations) were conducted using the Grams/386 software from Galactic Industries Corporation. The standard error of prediction (coefficient of variation) and coefficient of determination, R^2, were calculated for each of the models on each of the instruments. These values were then used to compare the models from the respective instruments. TABLE 4 summarizes the standard error of prediction (SEP) and R^2 values obtained. Appendix A contains plots of all calibration results. Examination of these results shows that the calibration models for any given fuel property were essentially equivalent regardless of the instrument used.

TABLE 2. Fuel Analyses

Property	Test Method
Fuel Lubricity, Wear Scar Diameter, mm	High Frequency Reciprocating Rig (HFRR) (proposed ISO and ASTM test method)
Fuel Lubricity, Scuffing Load, g	U.S. Army Scuffing Load Wear Test (SLWT) (proposed ASTM test method)*
Wear Scar Diameter, mm	ASTM D 5001†, Ball-on-Cylinder Lubricity Evaluator (BOCLE)
Sulfur, mass%	ASTM D 4294
Aromatic Hydrocarbons, mono-, di-, tri-, and total, mass%	ASTM D 5186
Kinematic Viscosity at 40°C, mm^2/s	ASTM D 445
Cloud Point, °C	Automatic Tester
Freeze Point, °C	Automatic Tester
Pour Point, °C	ASTM D 97
Distillation, °C	ASTM D 86
Density at 15°C, g/mL	ASTM D 4052 and ASTM D 1298
Cetane Number	ASTM D 613
Steam Jet Gum, mg/100 mL	ASTM D 381
Carbon and Hydrogen Content, mass%	ASTM D 5291
Total Water, ppm	Karl Fischer Titration, ASTM D 1744
Flash Point, °C	ASTM D 93
Net Heat of Combustion, MJ/kg	ASTM D 240

* A more complete description of the U.S. Army Scuffing Load Wear Test can be found in Reference 25.

† Test methods beginning with D refer to ASTM standards found in Volume 5 of the Book of Standards.

Three properties (density, 50% boiling point, and aromatics content) were selected for the validation phase. (The calibration results for these three properties are highlighted in TABLE 4.) An NIR spectrum was collected for each of the validation fuels using each of the three instruments. Using the appropriate calibration models from each instrument, the fuel property values were calculated from the NIR spectra of the validation fuels. The predicted fuel property values were then plotted against the property values measured in the laboratory. The R^2 values, based on a standard least squares fit of the data, are given in TABLE 5. The plots are presented in Appendix B. As with the calibration results, there were essentially no differences between instruments regarding the validation results.

TABLE 3. Descriptive Statistics for Test Fuels

Property	Mean	Range	Maximum	Minimum	Median
Manual Density, g/L (D 1298)	0.844	0.0823	0.870	0.788	0.847
Automatic Density, g/L (D 4052)	0.845	0.0845	0.872	0.787	0.848
Flash Point, °C (D 93)	61.984	47.000	87.000	40.000	62.000
Cloud Point, °C (D 1500)	–16.744	60.200	–0.300	–60.500	–14.100
Freeze Point, °C (D 2386)	–13.259	61.800	2.400	–59.400	–10.600
Pour Point, °C (D 97)	–30.767	69.000	–6.000	–75.000	–27.000
Viscosity at 40°C, mm^2/s (D 445)	2.493	2.790	3.930	1.140	2.545
Initial Boiling Point, °C (D 86)	175.680	75.300	214.000	138.700	176.450
10% Recovered	212.177	98.000	256.400	158.400	213.700
50% Recovered	258.001	114.800	297.000	182.200	262.650
90% Recovered	312.293	116.700	340.000	223.300	317.000
95% Recovered	326.870	128.000	359.100	231.100	331.150
End Point	341.100	134.400	375.500	241.100	344.900
Cetane Number (D 613)	48.713	24.400	61.300	36.900	48.400
Carbon Content, mass% (D 5291)	86.538	2.870	87.610	84.740	86.600
Hydrogen Content, mass% (D 5291)	13.089	2.040	14.330	12.290	13.050
Carbon/Hydrogen	6.616	1.146	7.097	5.951	6.638
Heat of Combustion, MJ/kg (D 240)	42.683	1.159	43.458	42.299	42.662
Steam Jet Gum, mg/100 mL (D 381)	6.564	202.200	202.200	0.000	4.700
Total Water, ppm (D 1744)	59.874	97.000	122.000	25.000	59.000
Aromatics, mass%					
Mono-, (D 5186)	24.286	29.900	38.900	9.000	25.000
Di-, (D 5186)	5.791	12.200	12.800	0.600	5.800
Tri-, (D 5186)	1.101	3.400	3.400	0.000	1.100
Total Aromatics, mass% (D 5186)	31.173	36.500	47.200	10.700	32.400
Total Sulfur, mass% (D 4294)	0.0334	0.390	0.400	0.01000	0.0300
Fuel Lubricity, Wear Scar Diameter, mm (HFRR)	0.262	0.610	0.740	0.130	0.230
Fuel Lubricity, Scuffing Load, g (SLWT)	3155.814	4400.000	5400.000	1000.000	3150.000
Wear Scar Diameter, mm (D 5001, BOCLE)	0.579	0.360	0.800	0.440	0.580

TABLE 4. Results of Instrument Calibrations

Component	Standard Error of Prediction			R^2		
	Bran & Luebbe	Brimrose	IFS	Bran & Luebbe	Brimrose	IFS
API Gravity, (D 1298)	0.39949	0.43669	0.37153	0.97826	0.97402	0.98119
Density, g/mL (D 1298)	0.0019638	0.0021287	0.0018974	0.97853	0.97476	0.97995
API Gravity, (D 4052)	0.36292	0.40268	0.35143	0.98237	0.97829	0.98346
Density, g/mL (D 4052)	**0.0018185**	**0.0019638**	**0.0017674**	**0.98183**	**0.97881**	**0.98284**
Flash Point, °C (D 93)	6.0261	6.0502	5.9534	0.42408	0.42195	0.43753
Cloud Point, °C (D 1500)	4.5347	4.57	5.1308	0.75955	0.75554	0.69204
Freeze Point, °C (D 2386)	4.6629	4.7407	5.0789	0.75231	0.74407	0.7063
Viscosity at 40°C, mm^2/s (D 445)	0.12482	0.13866	0.14935	0.9338	0.91833	0.90589
Boiling Point at 50% Distilled, °C (D 86)	**5.2821**	**5.4387**	**5.3055**	**0.92008**	**0.91514**	**0.91938**
Cetane Number (D 613)	2.3431	2.3288	2.3332	0.57179	0.57728	0.58127
Carbon, mass% (D 5291)	0.2971	0.28839	0.3041	0.48384	0.51363	0.45949
Hydrogen, mass% (D 5291)	0.099009	0.092481	0.097232	0.90065	0.91332	0.90418
Carbon/Hydrogen	0.048944	0.045407	0.046524	0.92364	0.93427	0.931
Heat of Comb., (D 240)						
MJ/kg	0.070759	0.070382	0.072452	0.82716	0.82896	0.8188
Btu/lb	30.256	30.074	30.949	0.82906	0.83108	0.82116
Gums, mg/100 mL (D 381)	6.6754	6.6714	6.656	0.0013694	0.0000785	0.0033346
Total Aromatics, mass% (D 5186)	**1.278**	**1.3142**	**1.3196**	**0.95762**	**0.95518**	**0.95485**
Sulfur, mass% (D 4294)	0.016234	0.01711	0.01766	0.31841	0.20572	0.1541

TABLE 5. R^2 Values for Validation Results

Property	Bran & Luebbe	Brimrose	IFS
Density, g/mL (D 4052)	0.977058	0.974275	0.987715
Boiling Pt. at 50%, °C (D 86)	0.915609	0.902124	0.936409
Total Aromatics, mass% (D 5186)	0.986774	0.985630	0.991295

Ease of use was not considered a deciding factor in the purchase decision of the instruments because each possessed good and poor features. Additionally, each of the three instrument suppliers indicated a willingness to consider small design changes to better meet Army needs.

Since there were no statistical differences in the results from the three instruments, the selection and purchase of NIR spectrometers for the follow-on work were based on instrument price. The quoted purchase prices for the Brimrose and Infrared Fiber Systems (IFS) instruments were similar. The quoted purchase price for the Bran & Luebbe instrument, however, was approximately 1.6 to 2.3 times greater than those for the other instruments. These prices were exclusive of computer costs, which were essentially equivalent for all three spectrometers.One other factor which entered into the purchase decision was a suggestion from the contract monitor that it would be beneficial to the overall program to include instruments from more than one supplier in the follow-on purchase.

For these reasons, six new AOTF-NIR instruments were purchased: three from Brimrose and three from IFS. Each of the instrument suppliers was also contacted prior to the purchase to discuss possible design changes as well as price discounts for multiple purchases.

V. SUMMARY AND FUTURE WORK

Three AOTF-NIR spectrometers were purchased from three instrument manufacturers. The instruments were comparatively evaluated to determine the best source for purchase of additional spectrometers.

Based on the preliminary evaluation results, six additional spectrometers were purchased: three instruments each were purchased from two of the original instrument manufacturers. Four hundred thirty fuel samples were then collected and analyzed for several common fuel properties.

The following work is planned:

- Optimize instrument calibrations for each of the fuel properties. This will involve use of alternative calibration techniques such as locally weighted regression.

- Obtain and analyze gasoline samples for calibration of the instruments.

- Evaluate methods for calibration transfer between the spectrometers.

- Conduct field trials at selected military installations with fully calibrated spectrometers.

- Develop and submit to ASTM for standardization proposed methods for the analysis of diesel fuels and kerosenes using NIR spectrometers.

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APPENDIX A

Calibration Results

Definition of Terms

The following definitions apply to the terms found in Appendix A:

Factor – Factors are mathematical "spectra" that are derived from the calibration spectra. These factors, when combined with the mean spectrum and some scalar values (scores), can be used to regenerate the original spectra. Selection of the proper number of factors is made based on how many factors are needed to sufficiently model the component of interest. Since the scores and factors are related to the concentration of the components, they can be used to measure the property values of an unknown sample. A calibration model consists of a collection of factors and scores.

Standard Error of Prediction (Cross-Validation), SEP (CV) – During the calibration process, one sample is removed from the calibration set and the remaining samples are used to build a calibration model. The calibration model is then used to measure the component values(s) for the sample that was removed, treating it as if it were an unknown. This process, known as cross-validation (CV), is carried out until each sample has been left out once. SEP (CV) is a measure of the performance of the calibration model and is given in the same units as the component being modeled.

Total Error, TE – The Total Error is the sum of the absolute value of the error for all of the samples. The error is determined as the difference between the known value and the measured value.

Root Mean Squared Difference, RMSD – The Root Mean Squared Difference gives an indication of the average error in the calibration model for a given component.

Squared Correlation Coefficient, R^2 – The Squared Correlation Coefficient indicates the quality of fit of the predicted values to the known values for a given component. A value of 1 indicates a perfect match between the measured and known values.

H. Bjorsvik and H. Martens, "Data Analysis: Calibration of NIR Instruments by PLS Regression," in Handbook of Near-Infrared Analysis, Burns and Ciurczak, ed., p. 158, Marcel Dekker, New York, NY (1992).

Abbreviations

The following abbreviations are used in the predicted *vs.* actual plots in Appendix A:

Component Name	Abbreviation
Density, g/mL (D 1298)	d1298
Density, g/mL (D 4052)	d4052
Flash Point, °C (D 93)	flash
Cloud Point, °C (D 1500)	cloud
Freeze Point, °C (D 2386)	freeze
Pour Point, °C (D 97)	pour
Viscosity at 40°C, mm^2/s (D 445)	visc
Boiling Point at 50%, °C (D 86)	bp50
Cetane Number (D 613)	cetane
Carbon, mass% (D 5291)	c
Hydrogen, mass% (D 5291)	h
Carbon/Hydrogen	ch
Net Heat of Comb., MJ/kg (D 240)	mjkg
Gums, mg/100 mL (D 381)	gums
Total Water, ppm (D 1744)	water
Aromatics, Mono-, (D 5186)	mono
Aromatics, Di-, (D 5186)	di
Aromatics, Tri-, (D 5186)	tri
Total Aromatics, mass% (D 5186)	total
Sulfur, mass% (D 4294)	sulfur
Fuel Lubricity, mm (HFRR)	hfrr
Fuel Lubricity, g (SLWT)	scuff
Wear Scar Diameter, mm (D 5001, BOCLE)	bocle

Instrument: Bran & Luebbe

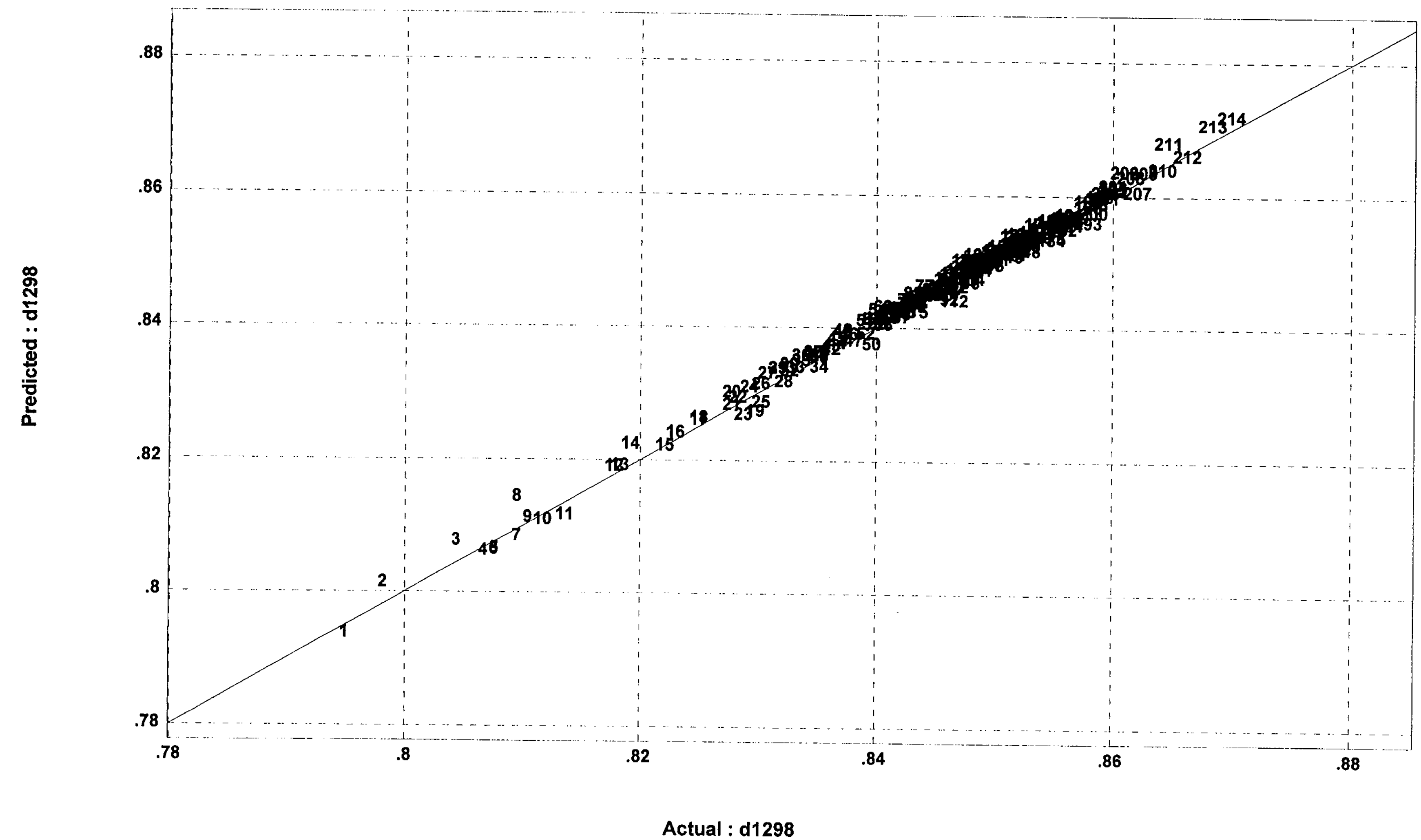
Calibration Summary: 23 components, 214 spectra, 401 points, 1 rotation sample, PLS1, mean centering

Component	Factor(recommended)	SEP(CV)	R^2
DENSITY (D 1298)	18	0.0011312	0.99234
DENSITY(D 4052)	19	0.000885	0.995405
FLASH	4	5.84	0.393074
CLOUD	13	4.3581	0.759682
FREEZE	12	4.8491	0.723783
POUR	13	5.6535	0.736876
VISCOSITY	18	0.11987	0.930608
BOILING PT @50%	18	3.9616	0.950576
CETANE	7	2.1861	0.573877
CARBON	4	0.27835	0.910966
HYDROGEN	7	0.090366	0.505413
CARBON/HYDROGEN	10	0.044013	0.934009
NET Ht. Comb. MJ/Kg	5	0.068153	0.825757
GUMS	1	14.567	0.0235143
WATER	1	14.364	0.0422699
AROMATICS, mono-	14	0.63095	0.980212
AROMATICS, di-	12	0.43956	0.952065
AROMATICS, tri-	13	0.28873	0.6819
TOTAL AROMATICS	16	0.58032	0.990761
SULFUR	17	0.017518	0.288277
HFRR	9	0.069191	0.566213
SLWT	4	639.07	0.18038
BOCLE	1	0.03203	0.000169532

TE: .0165093

R^2: .99234

RMSD: .00112855



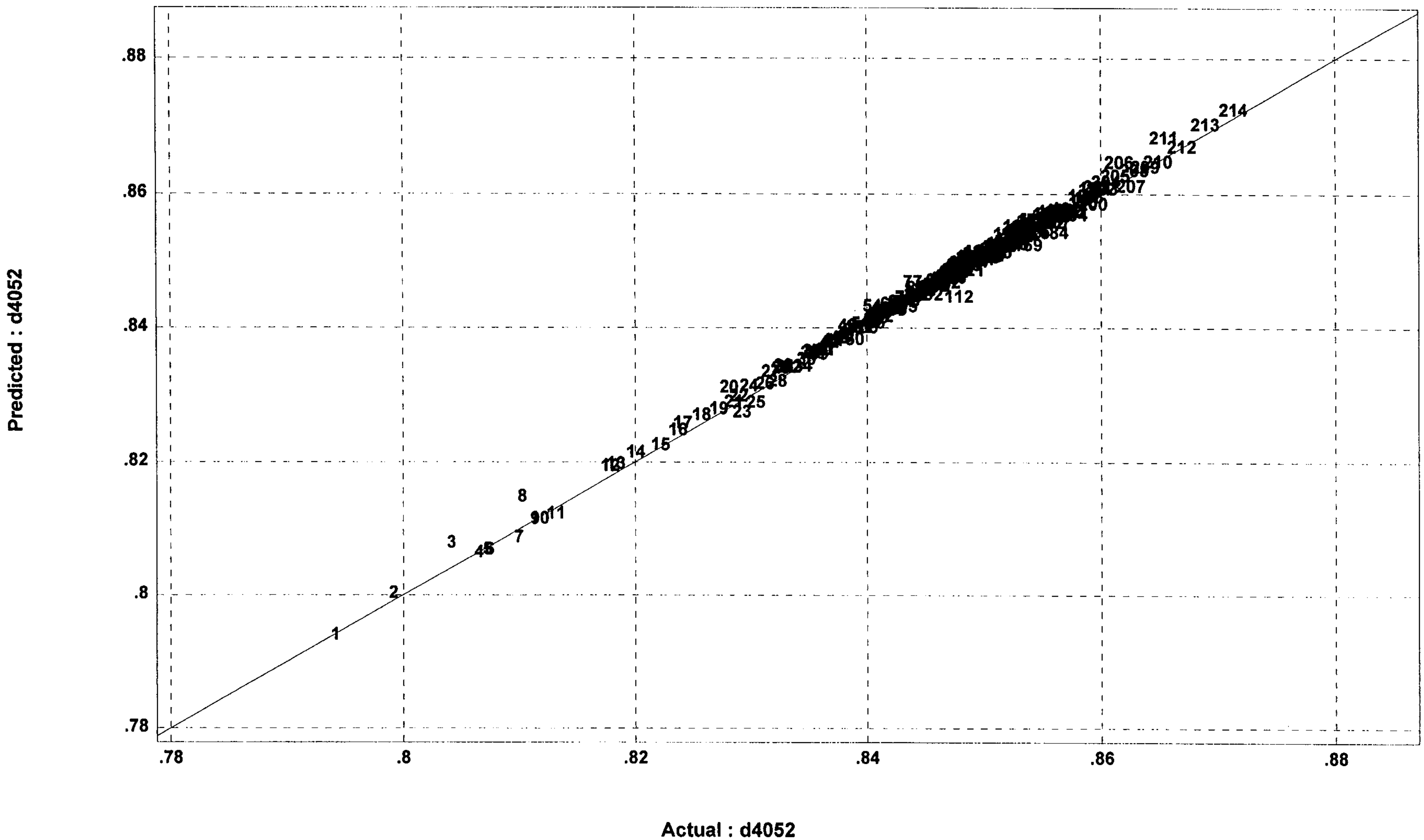
Total Factors: 18

Component: d1298

TE: .0129162

R^2: .995405

RMSD: .000882933



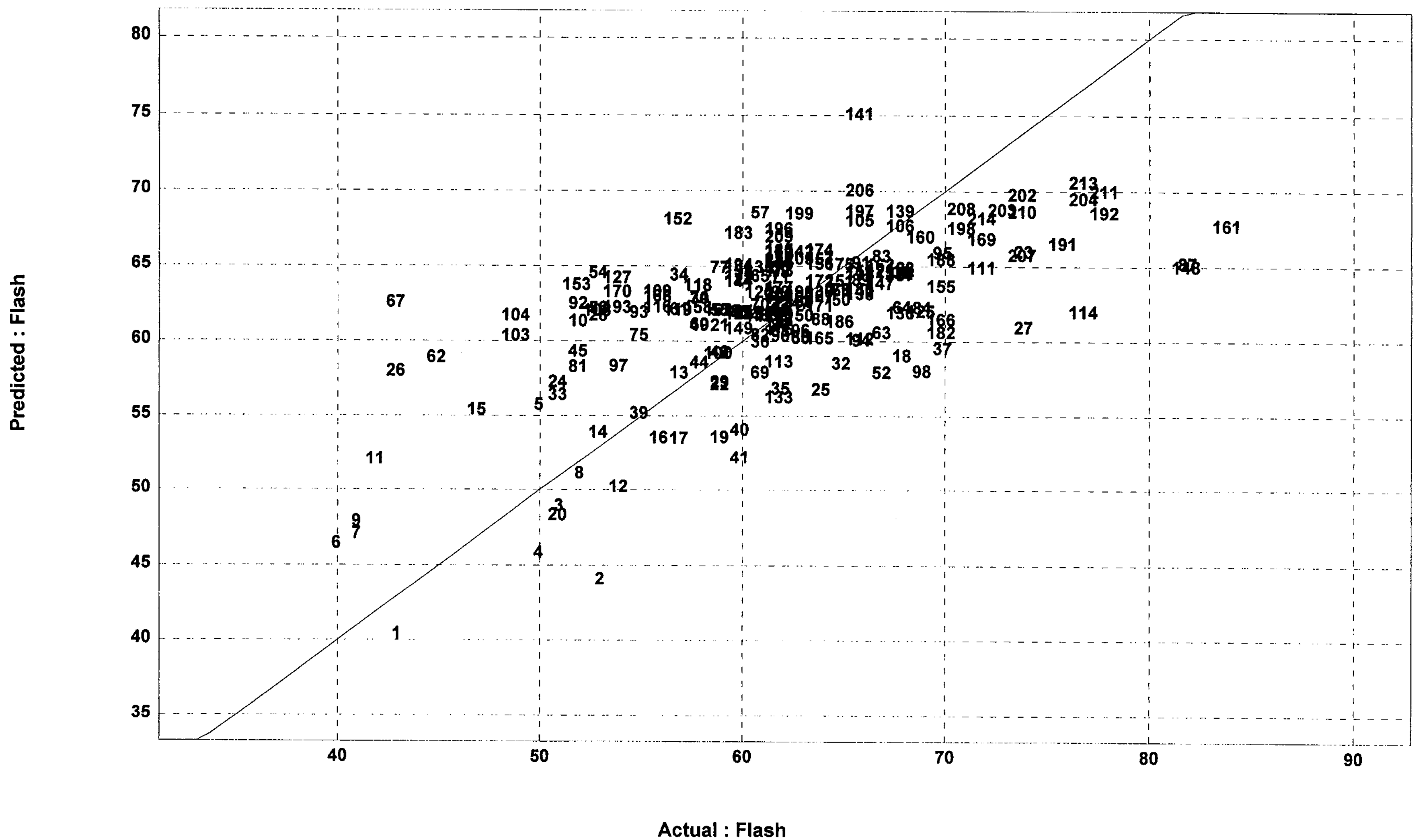
Total Factors: 19

Component: d4052

TE: 85.2326

R^2: .393074

RMSD: 5.82638



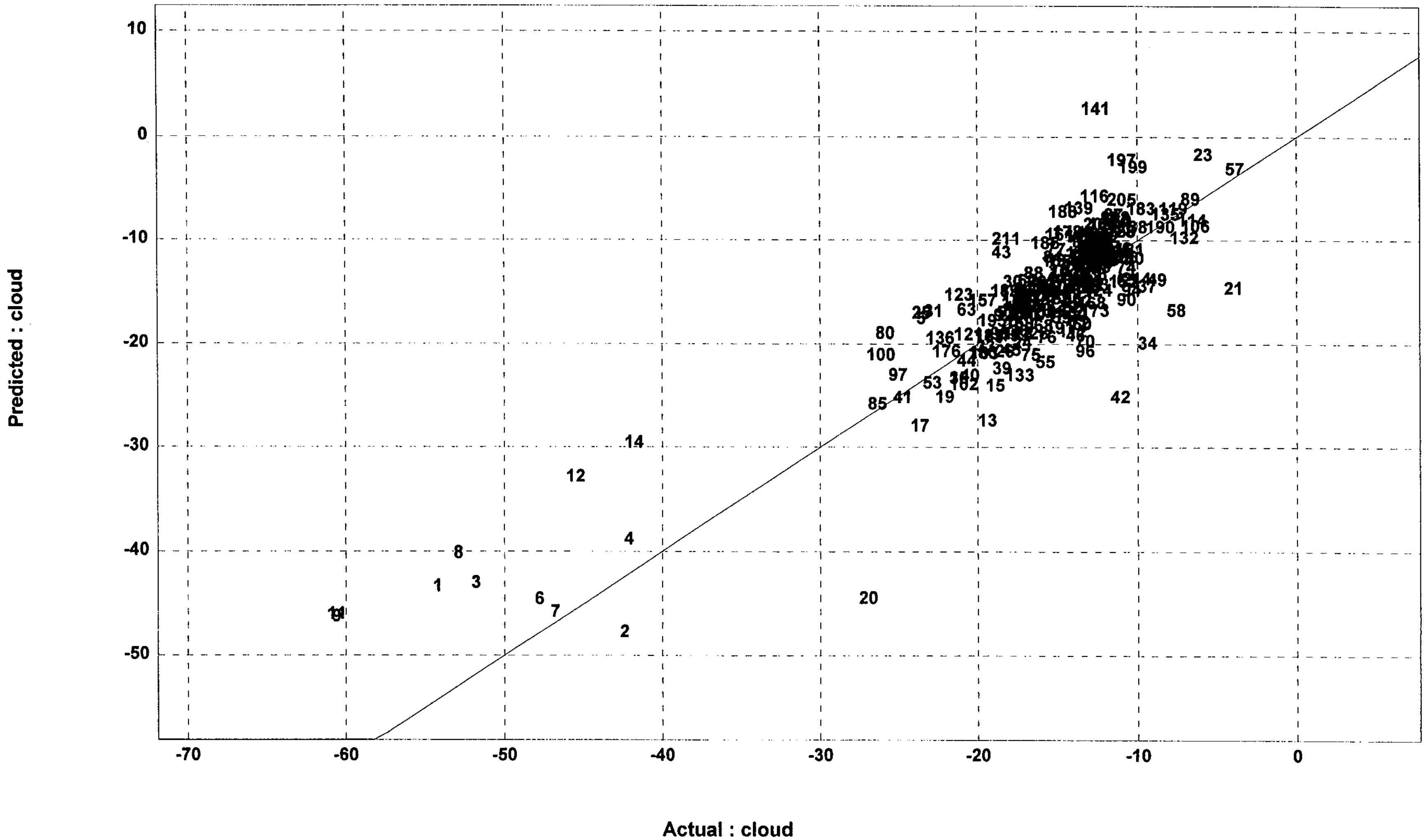
Total Factors: 4

Component: Flash

TE: 63.6039

R^2: .759682

RMSD: 4.34787



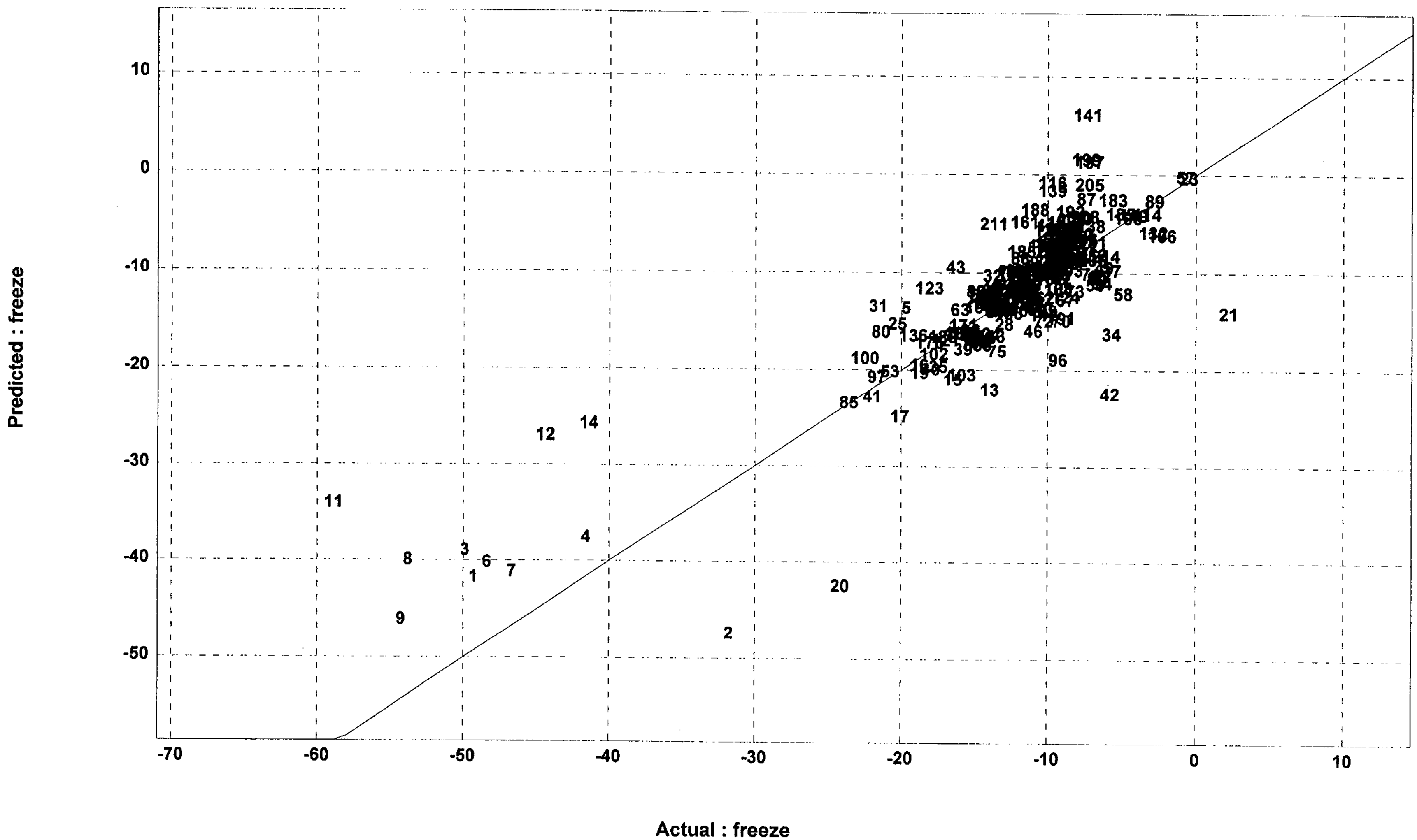
Total Factors: 13

Component: cloud

TE: 70.7706

R^2: .723783

RMSD: 4.83778



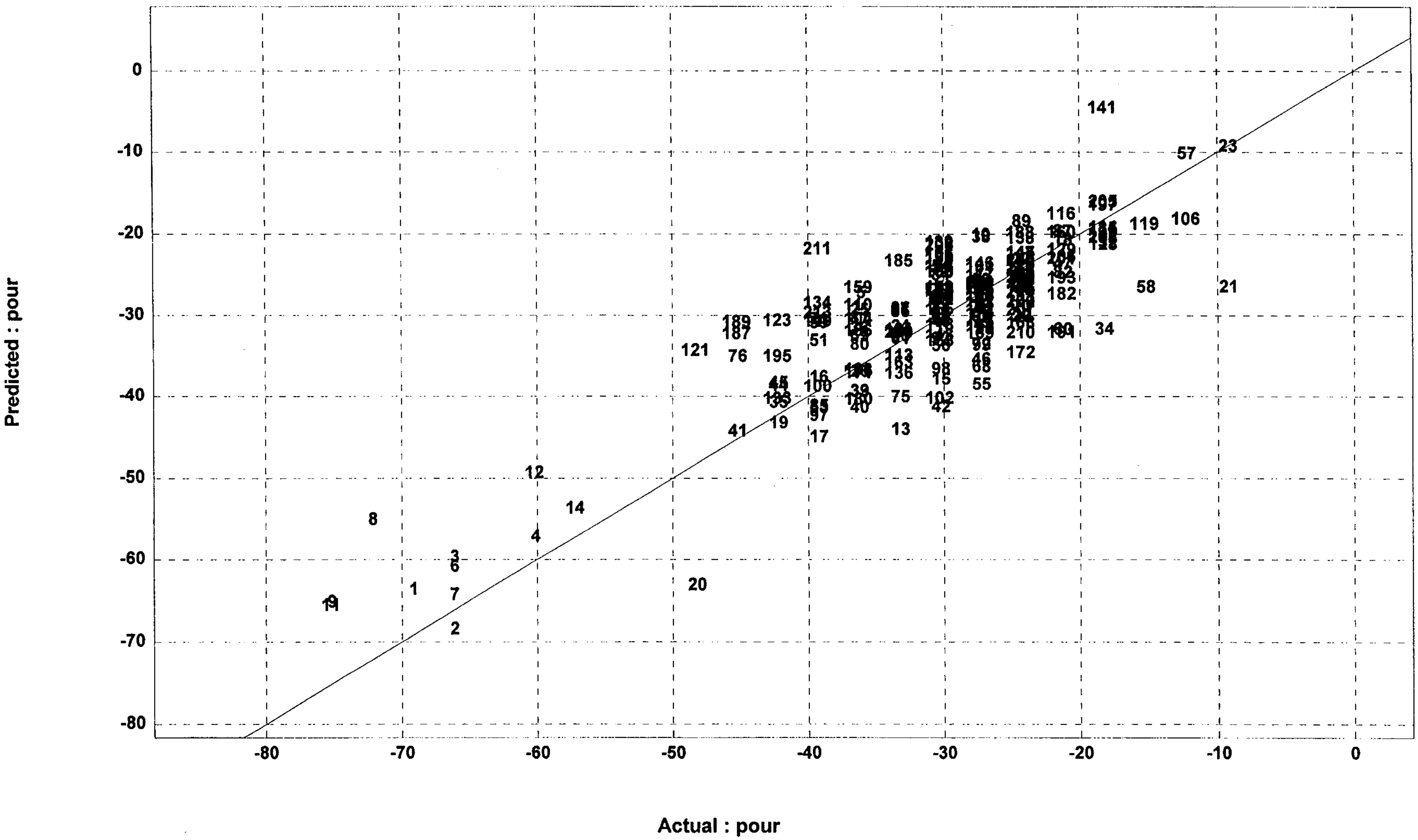
Total Factors: 12

Component: freeze

TE: 82.5107

R^2: .736876

RMSD: 5.64032



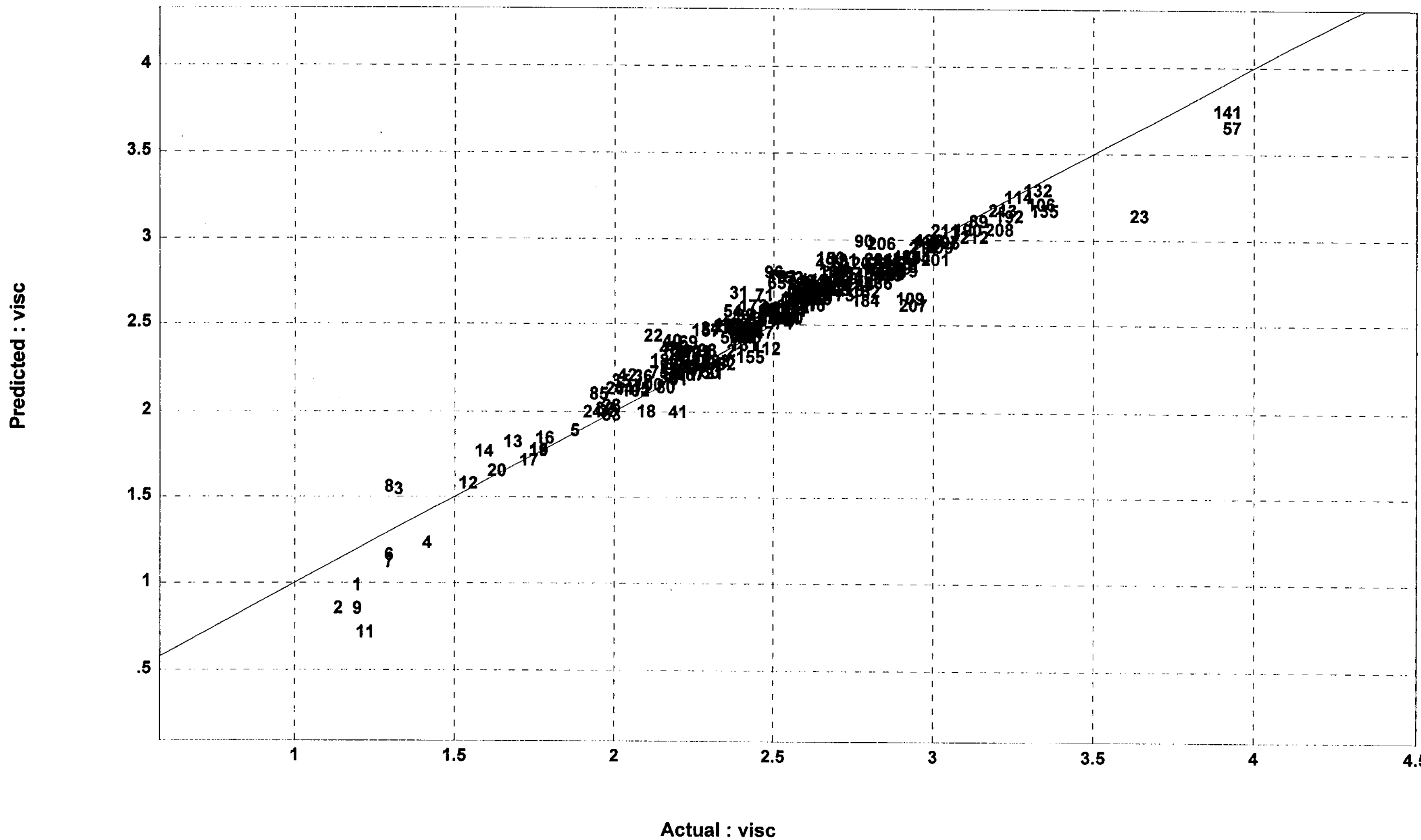
Total Factors: 13

Component: pour

TE: 1.74952

R^2: .930608

RMSD: .119595



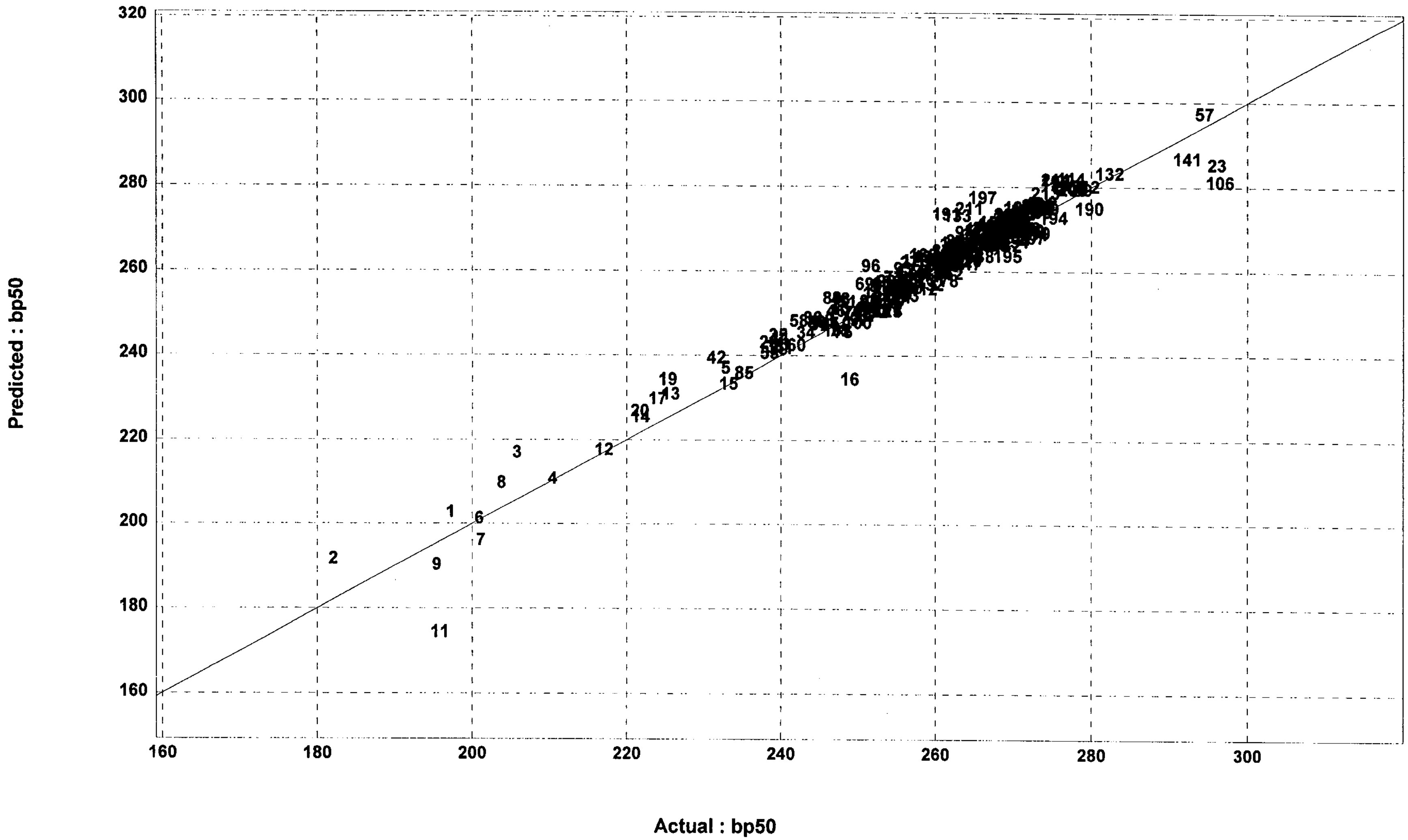
Total Factors: 18

Component: visc

TE: 57.8182

R^2: .950576

RMSD: 3.95237



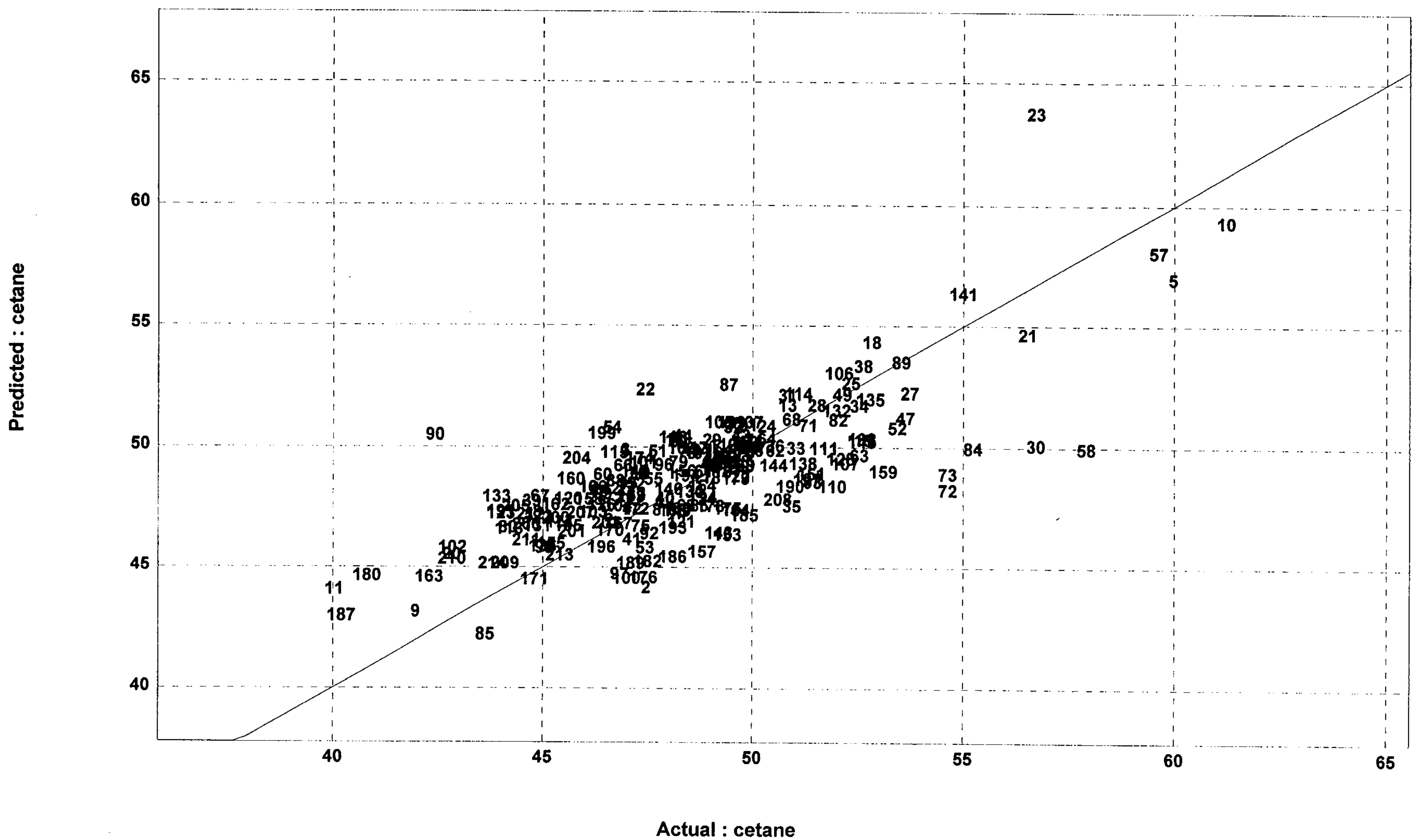
Total Factors: 18

Component: bp50

TE: 31.9056

R^2: .573877

RMSD: 2.18102



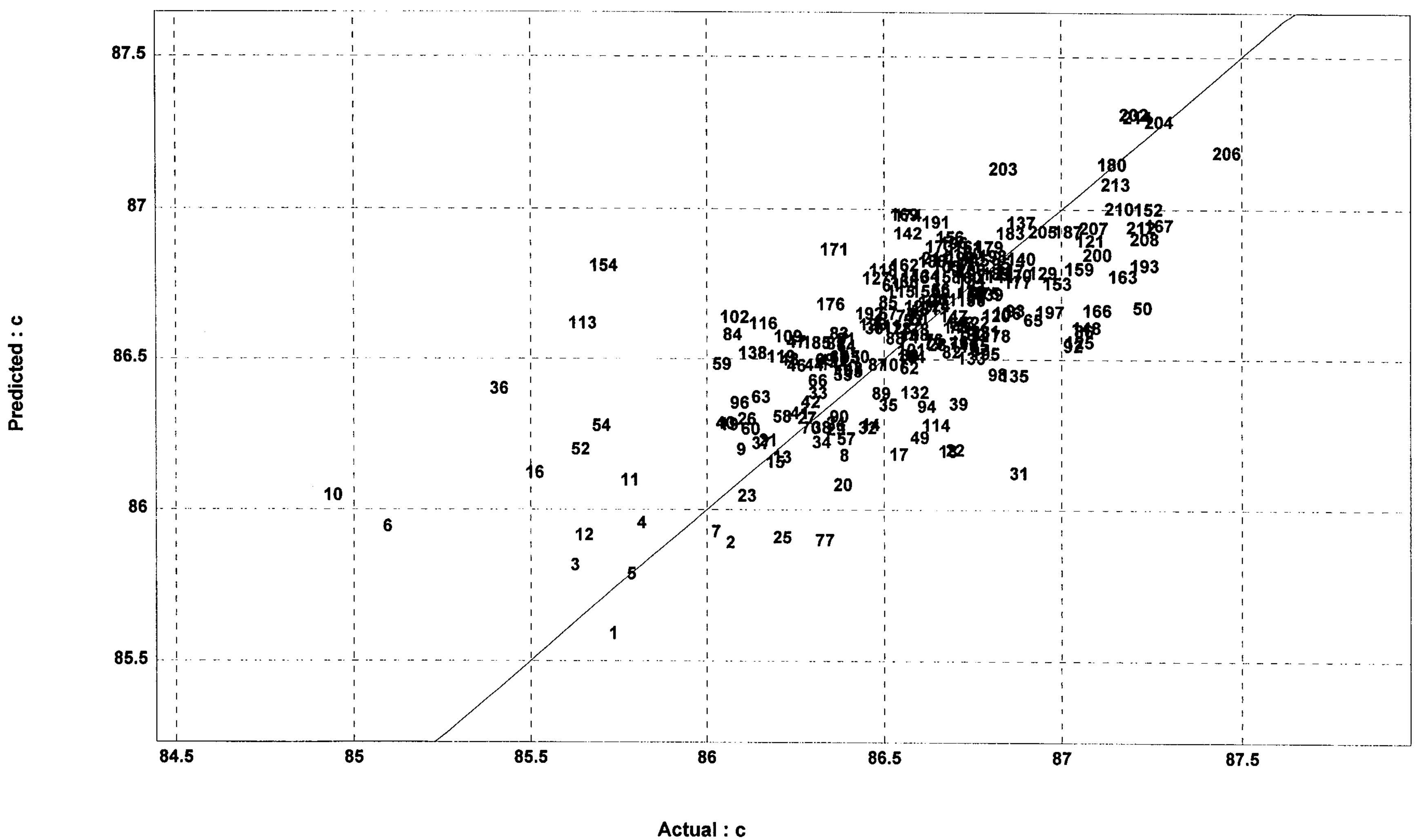
Total Factors: 7

Component: cetane

TE: 4.06242

R^2: .505413

RMSD: .277702



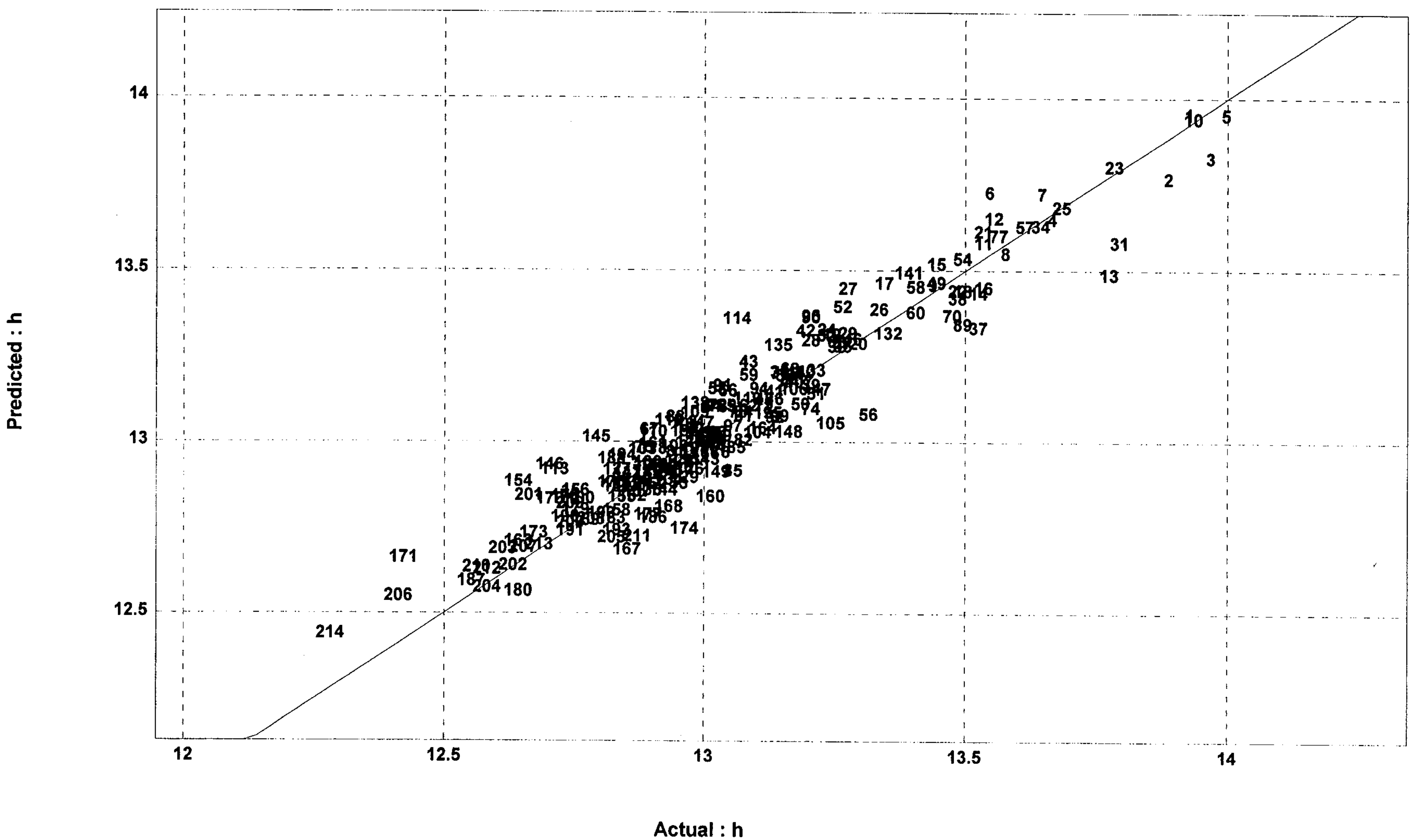
Total Factors: 4

Component: c

TE: 1.31885

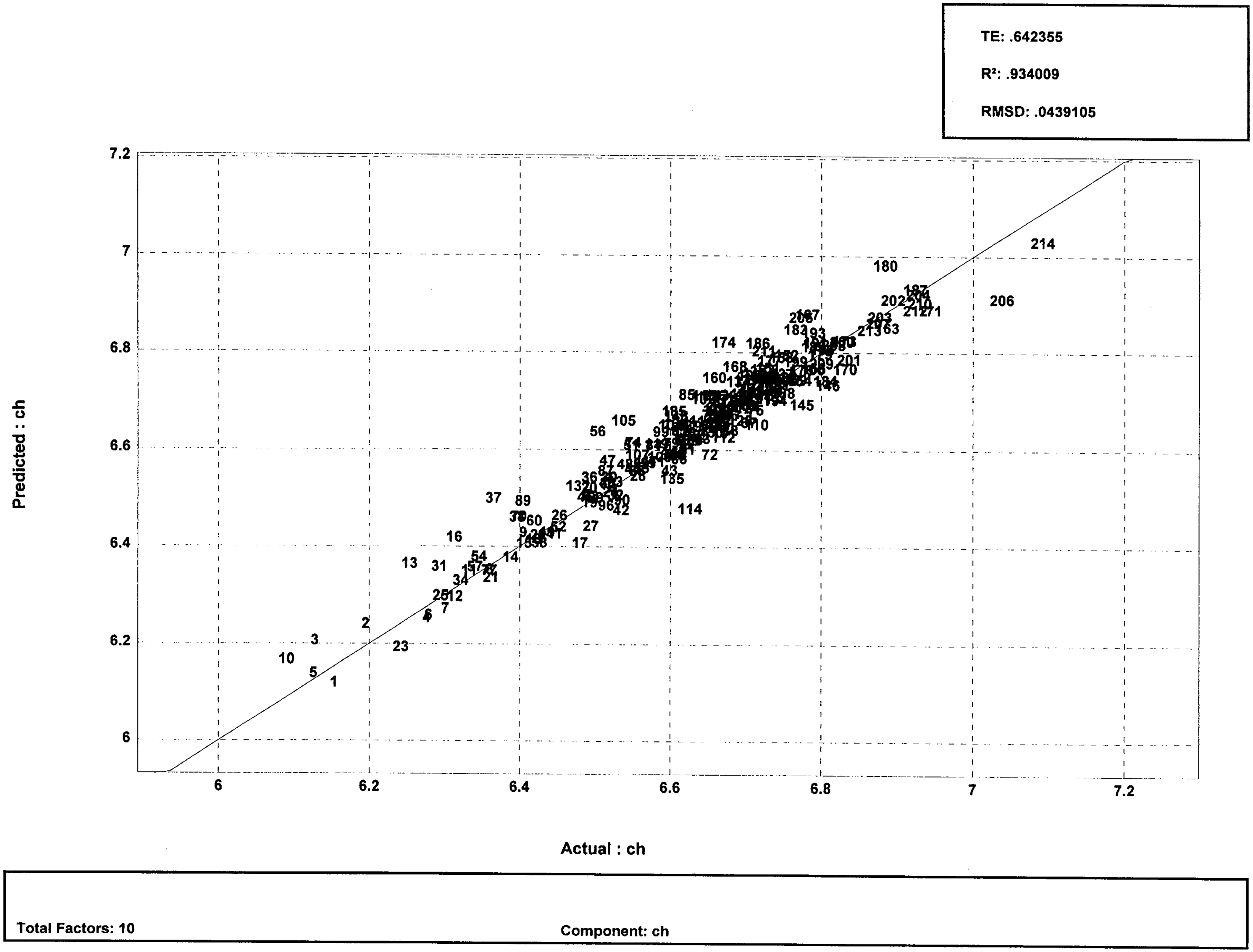
R²: .910966

RMSD: .0901549



Total Factors: 7

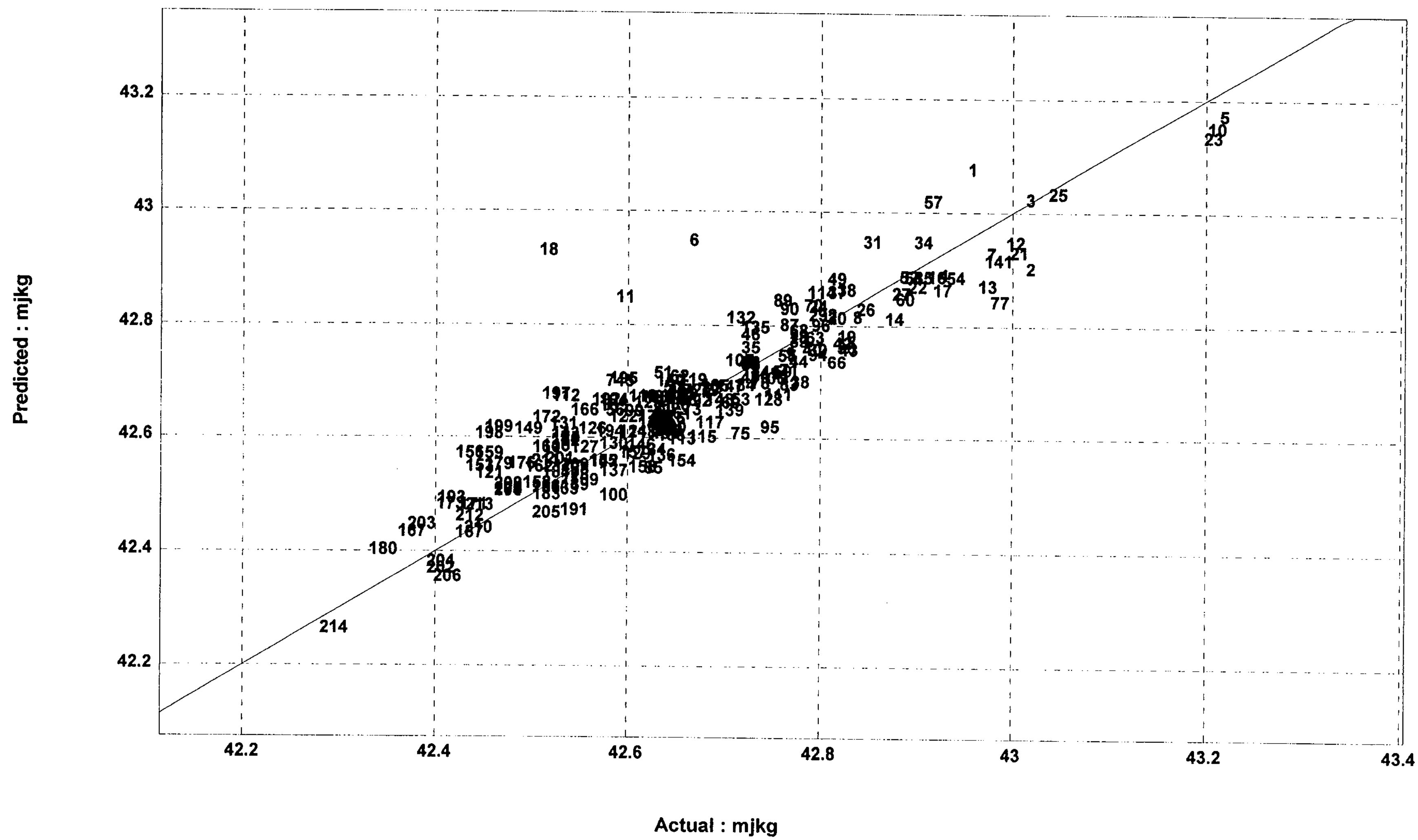
Component: h


TE: .642355
R²: .934009
RMSD: .0439105
Predicted : ch
Actual : ch
Total Factors: 10
Component: ch

TE: .994665

R^2: .825757

RMSD: .0679939



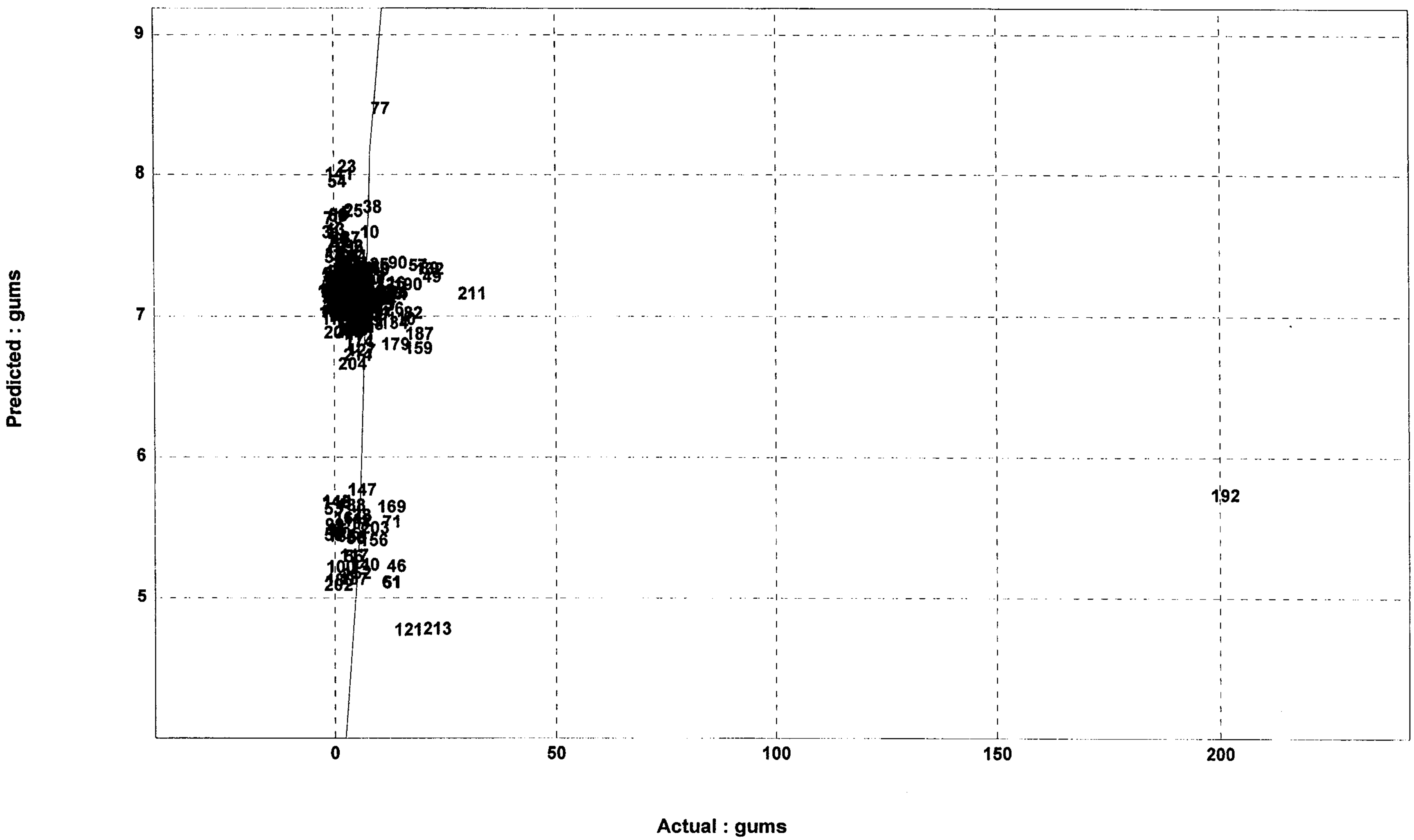
Total Factors: 5

Component: mjkg

TE: 212.606

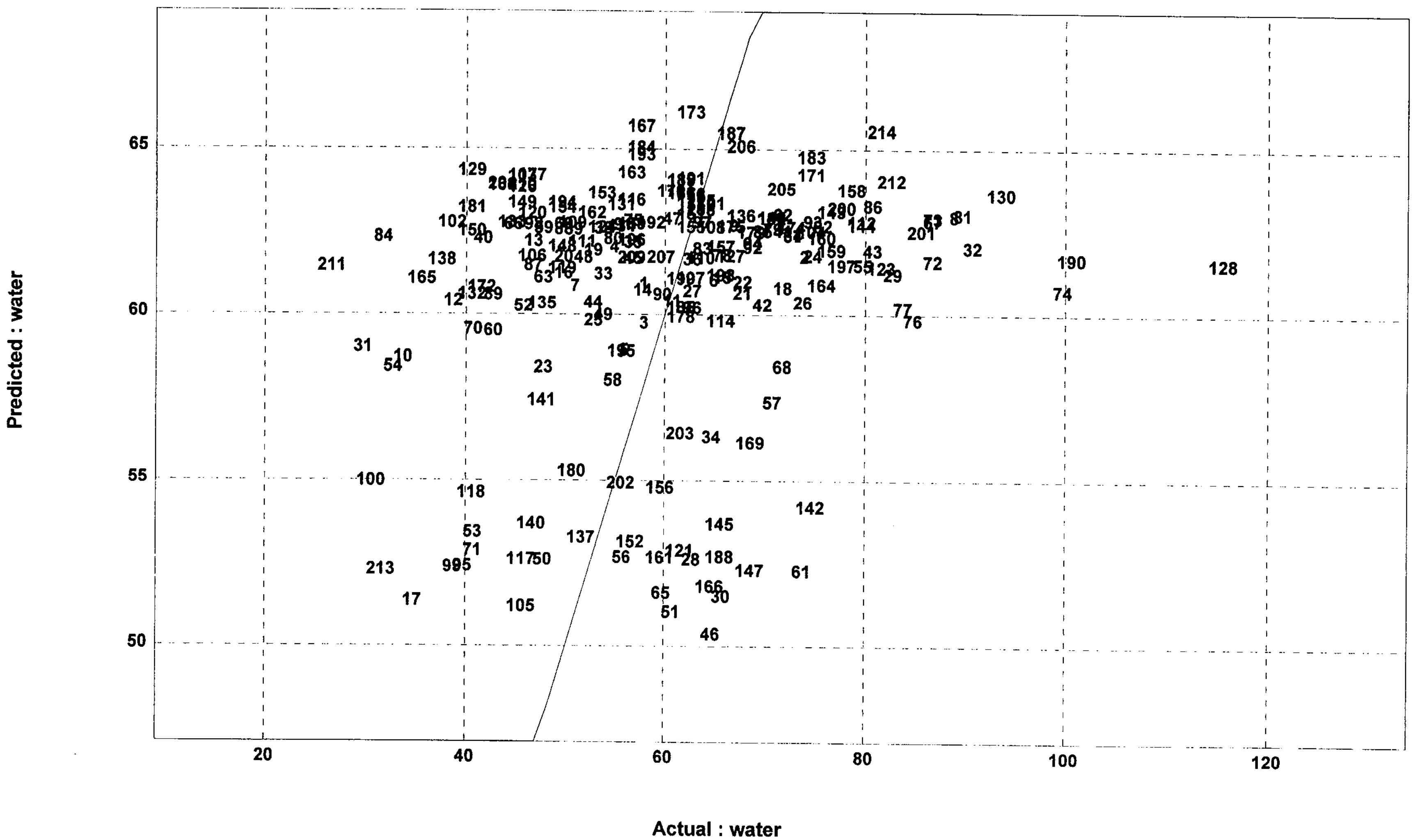
R^2: .0235143

RMSD: 14.5335



Total Factors: 1

Component: gums



TE: 209.643

R^2: .0422699

RMSD: 14.3309

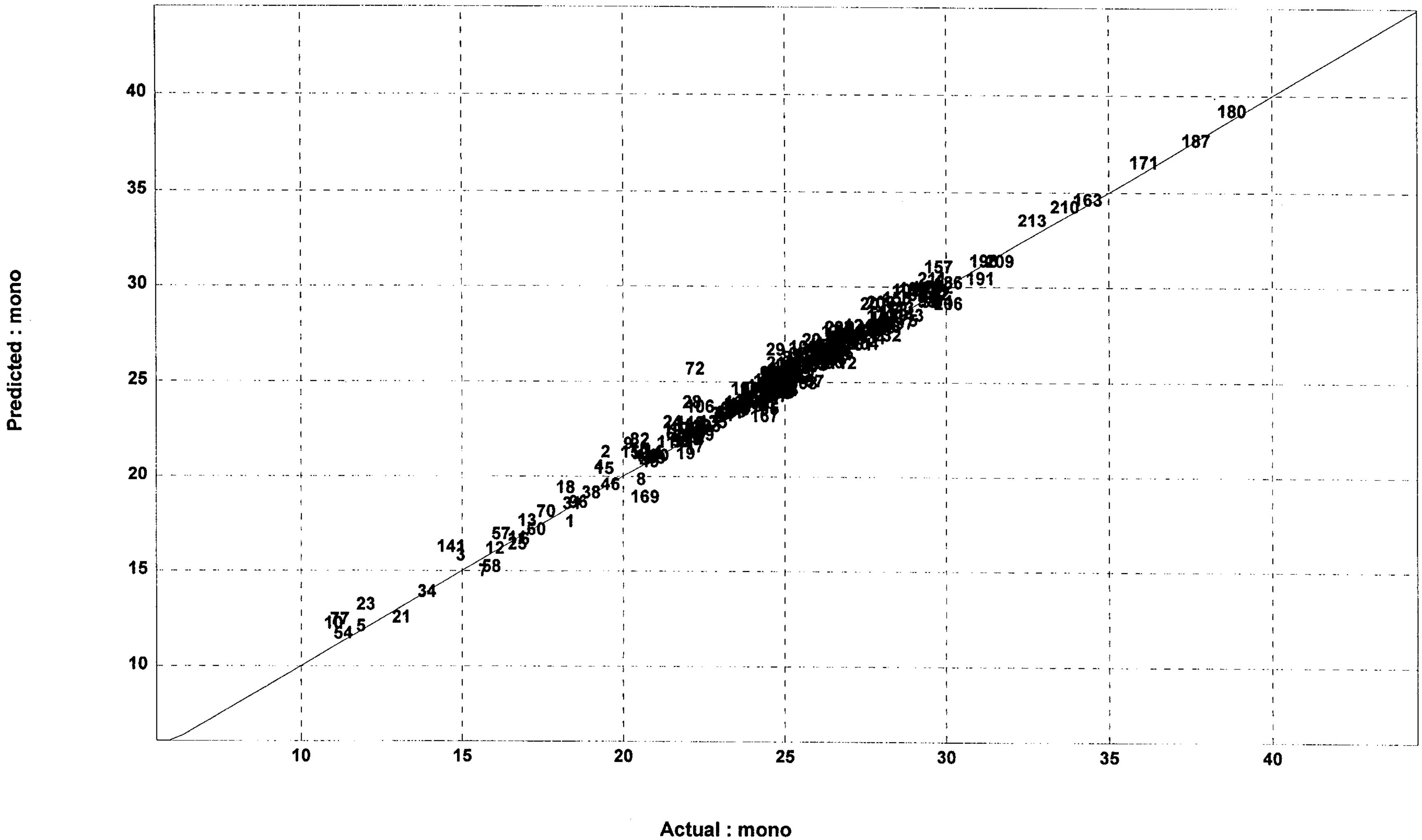
Total Factors: 1

Component: water

TE: 9.20843

R^2: .980212

RMSD: .629475



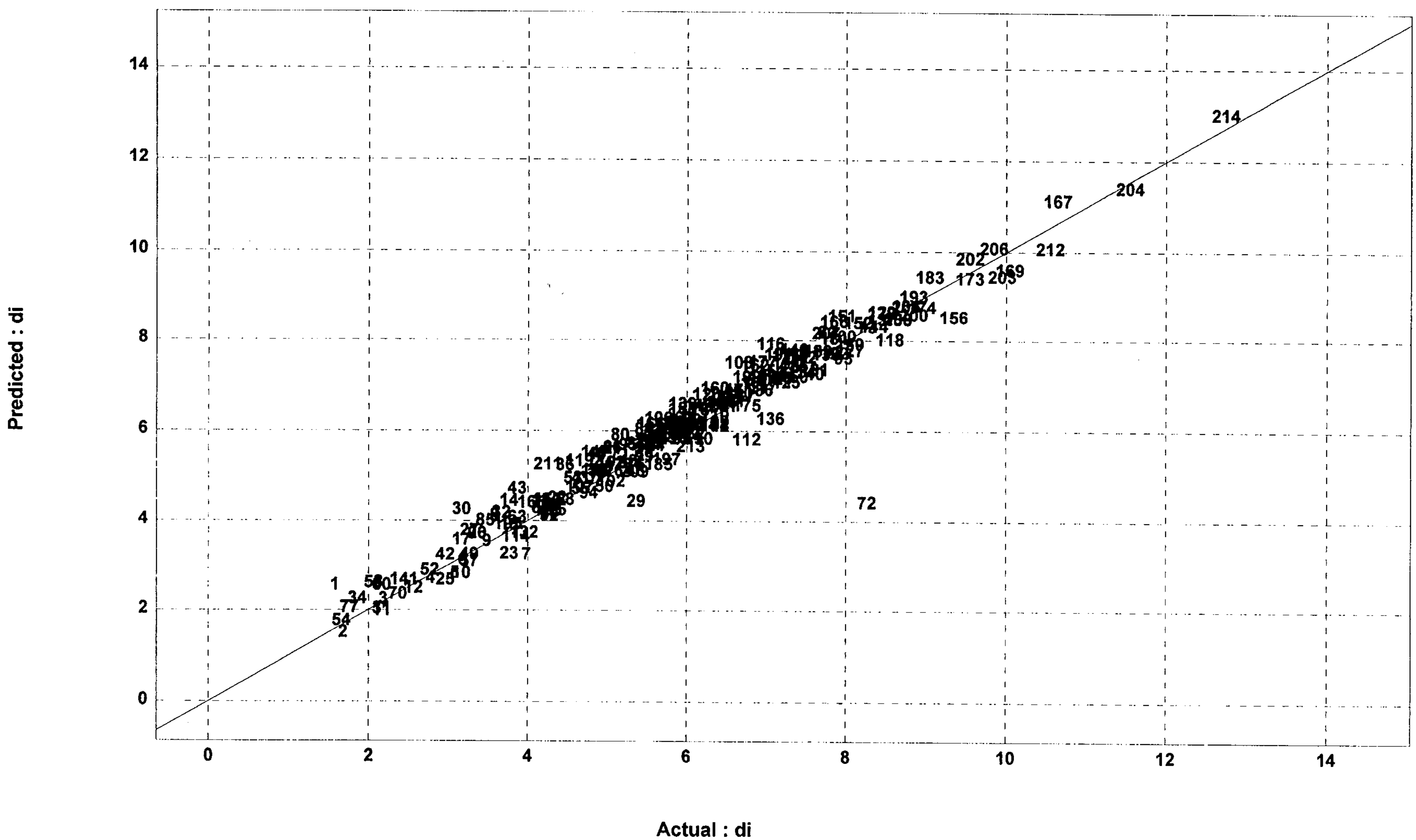
Total Factors: 14

Component: mono

TE: 6.41515

R²: .952065

RMSD: .43853



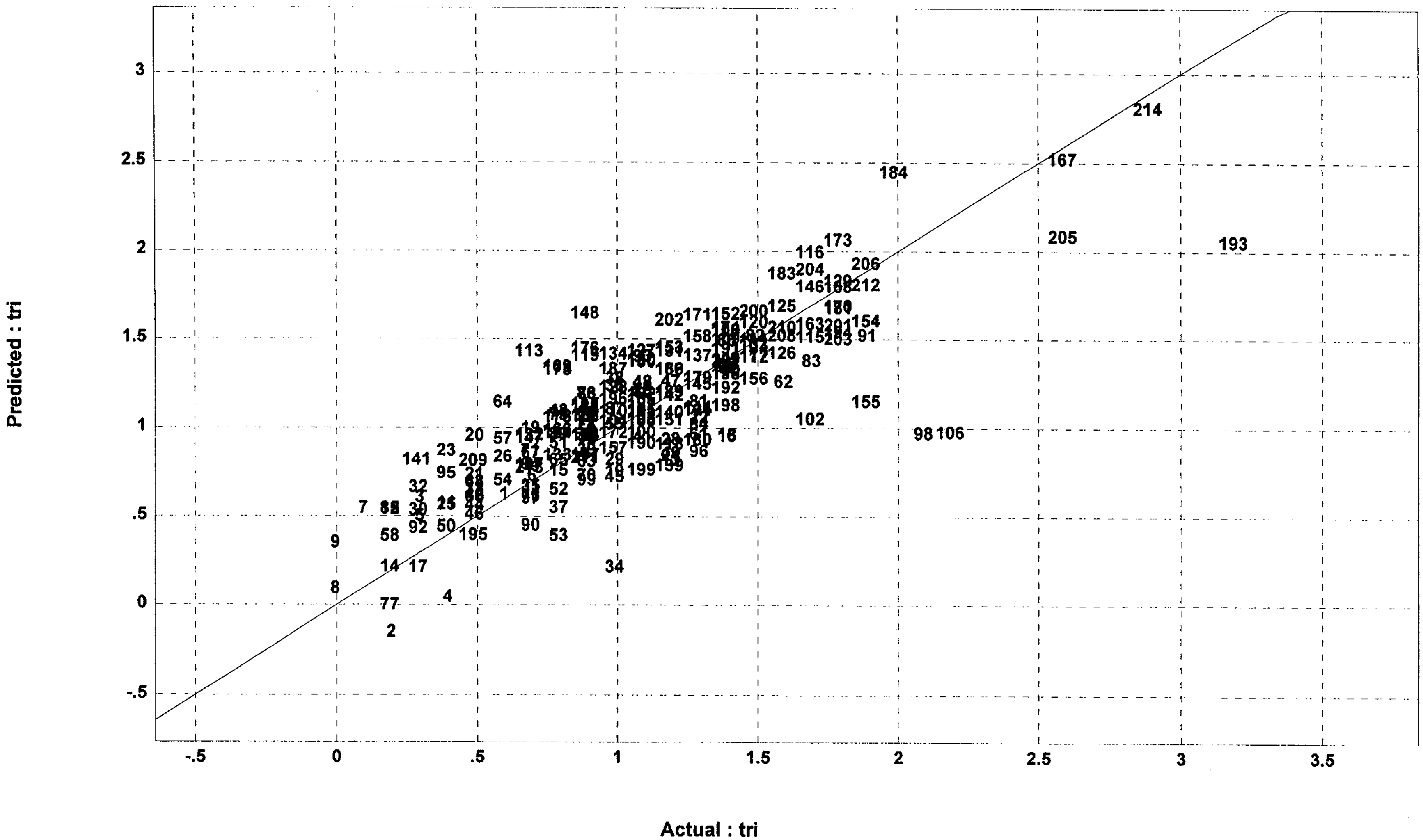
Total Factors: 12

Component: di

TE: 4.21383

R²: .6819

RMSD: .288052



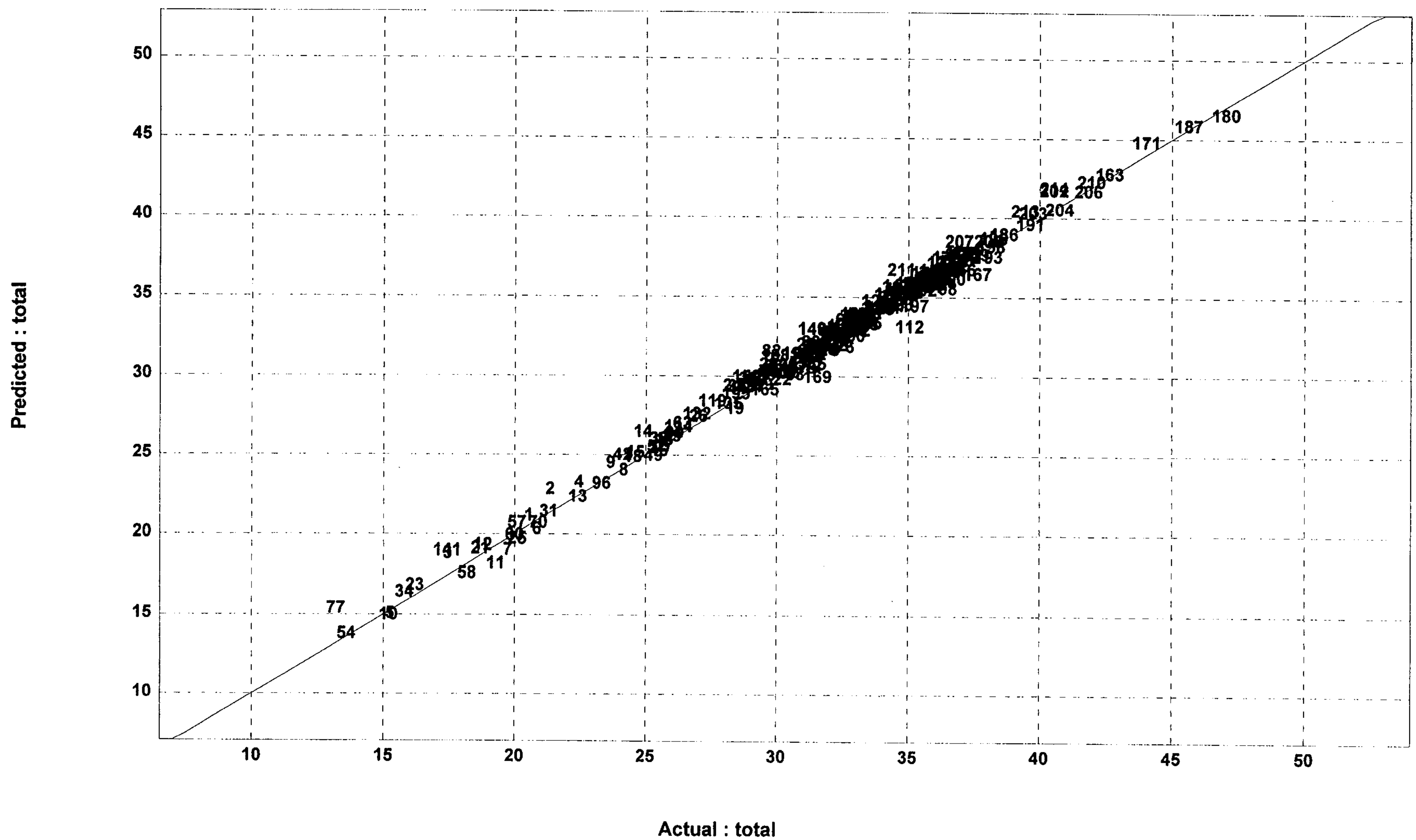
Total Factors: 13

Component: tri

TE: 8.46948

R^2: .990761

RMSD: .578962



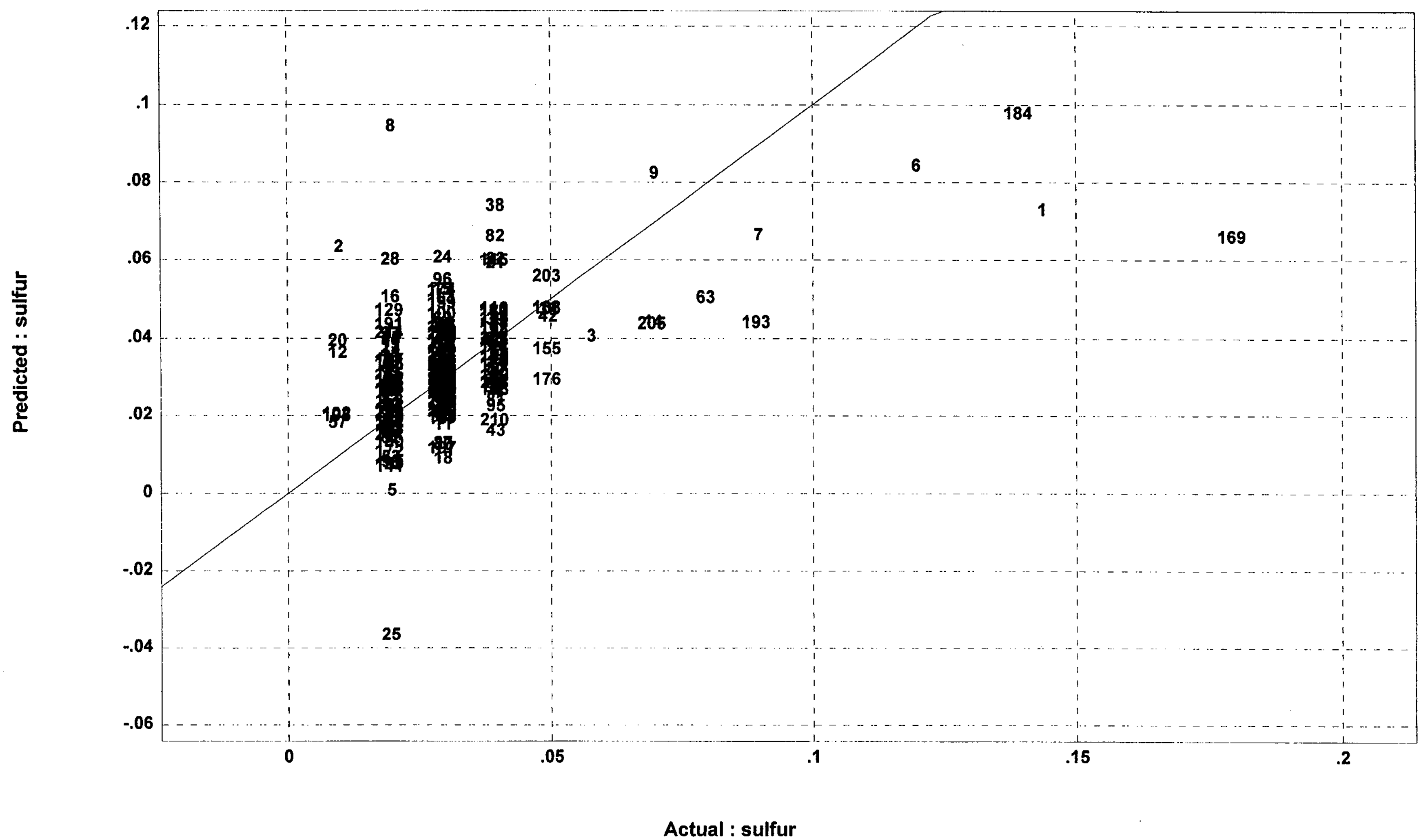
Total Factors: 16

Component: total

TE: .255674

R^2: .288277

RMSD: .0174775



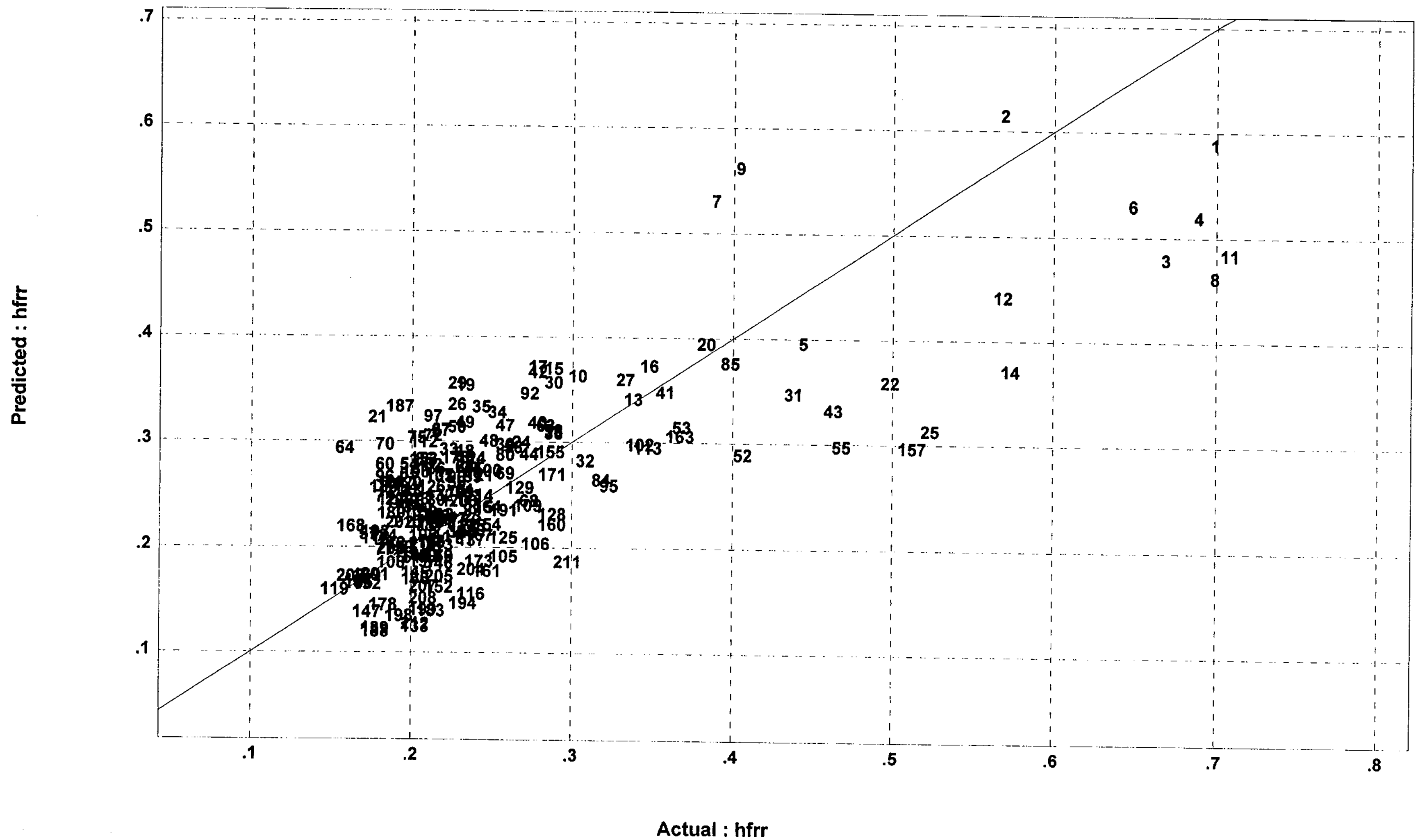
Total Factors: 17

Component: sulfur

TE: 1.00982

R^2: .566213

RMSD: .0690297



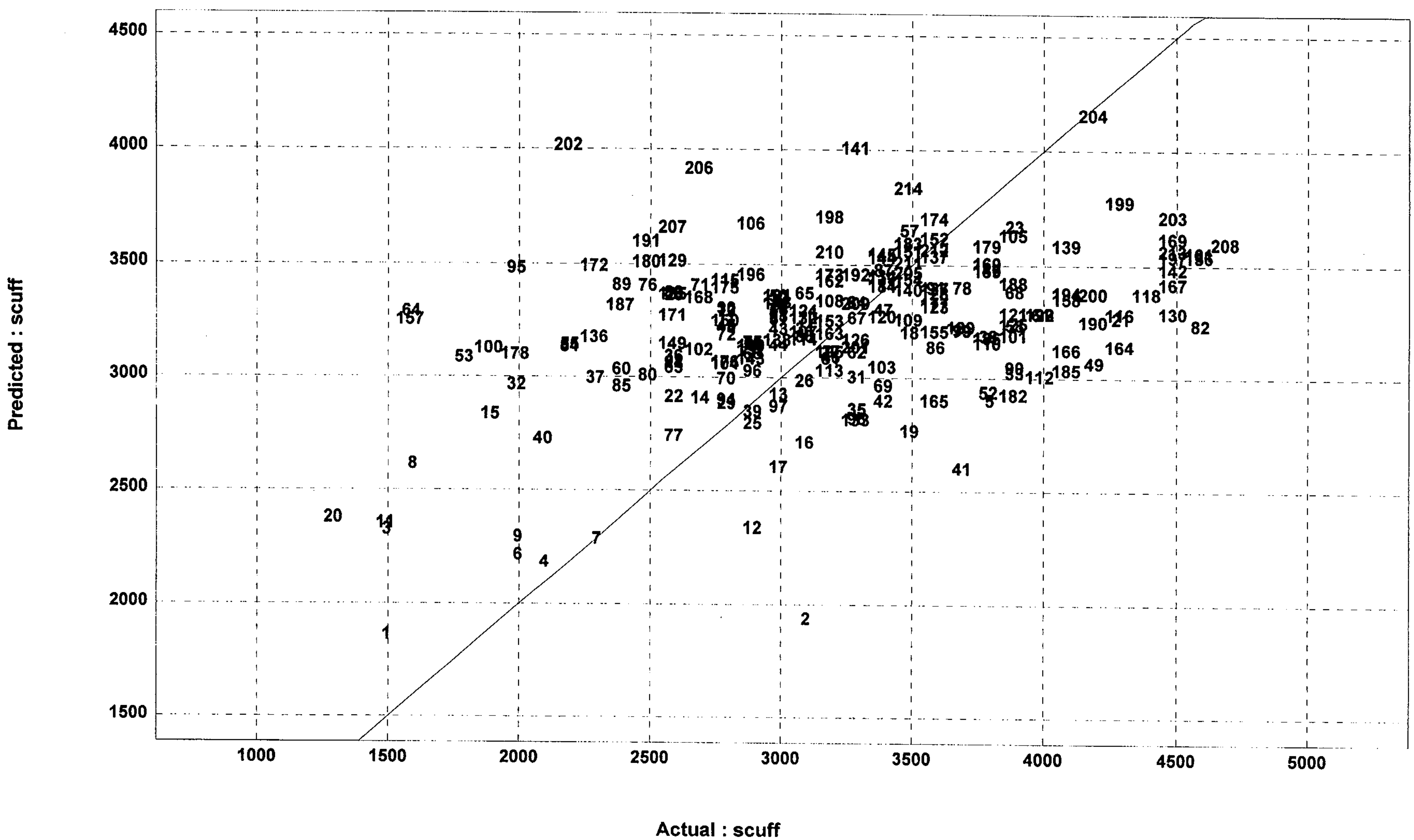
Total Factors: 9

Component: hfrr

TE: 9326.99

R^2: .18038

RMSD: 637.58



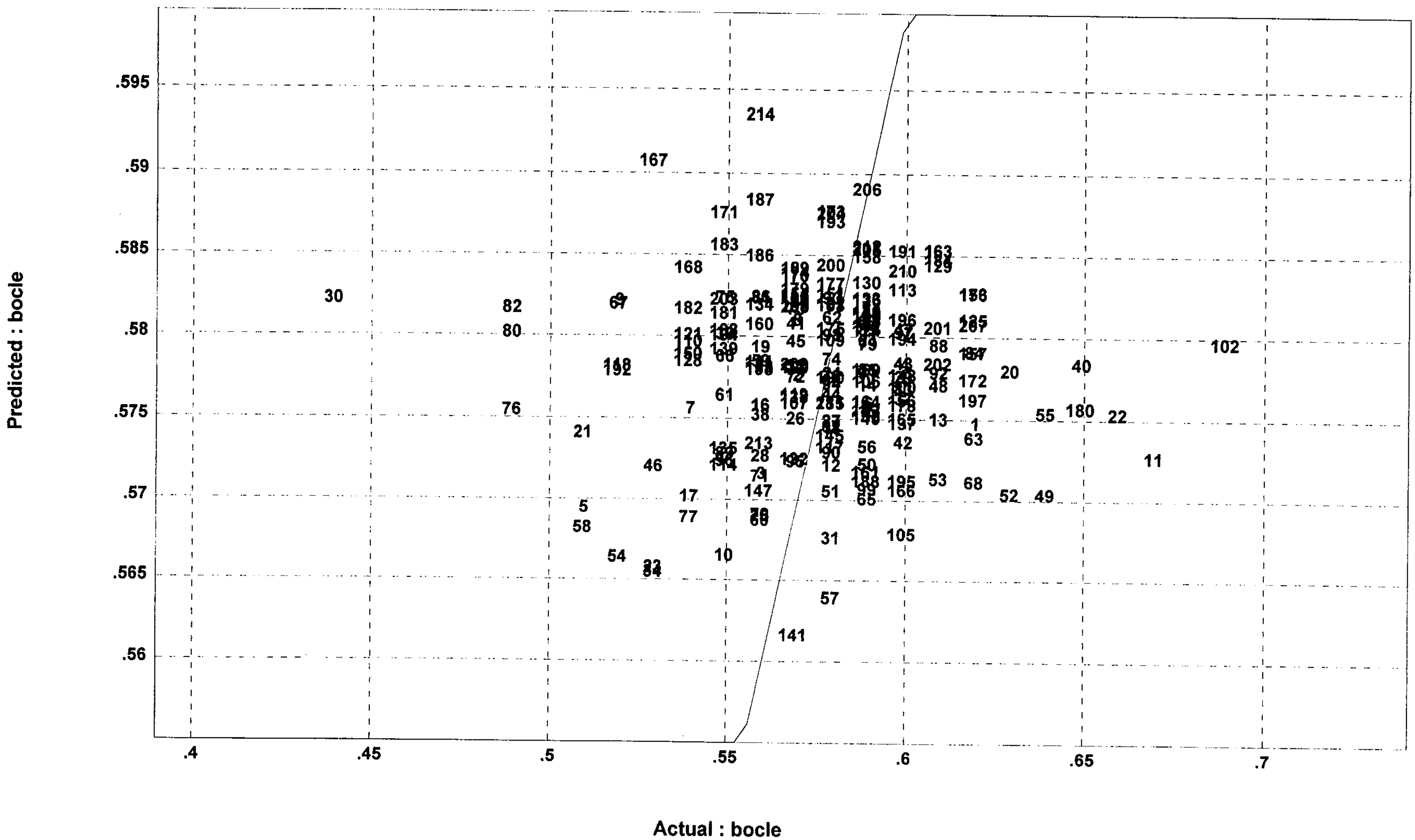
Total Factors: 4

Component: scuff

TE: .46746

R²: .000169532

RMSD: .0319549



Total Factors: 1

Component: bocle

Instrument: Brimrose

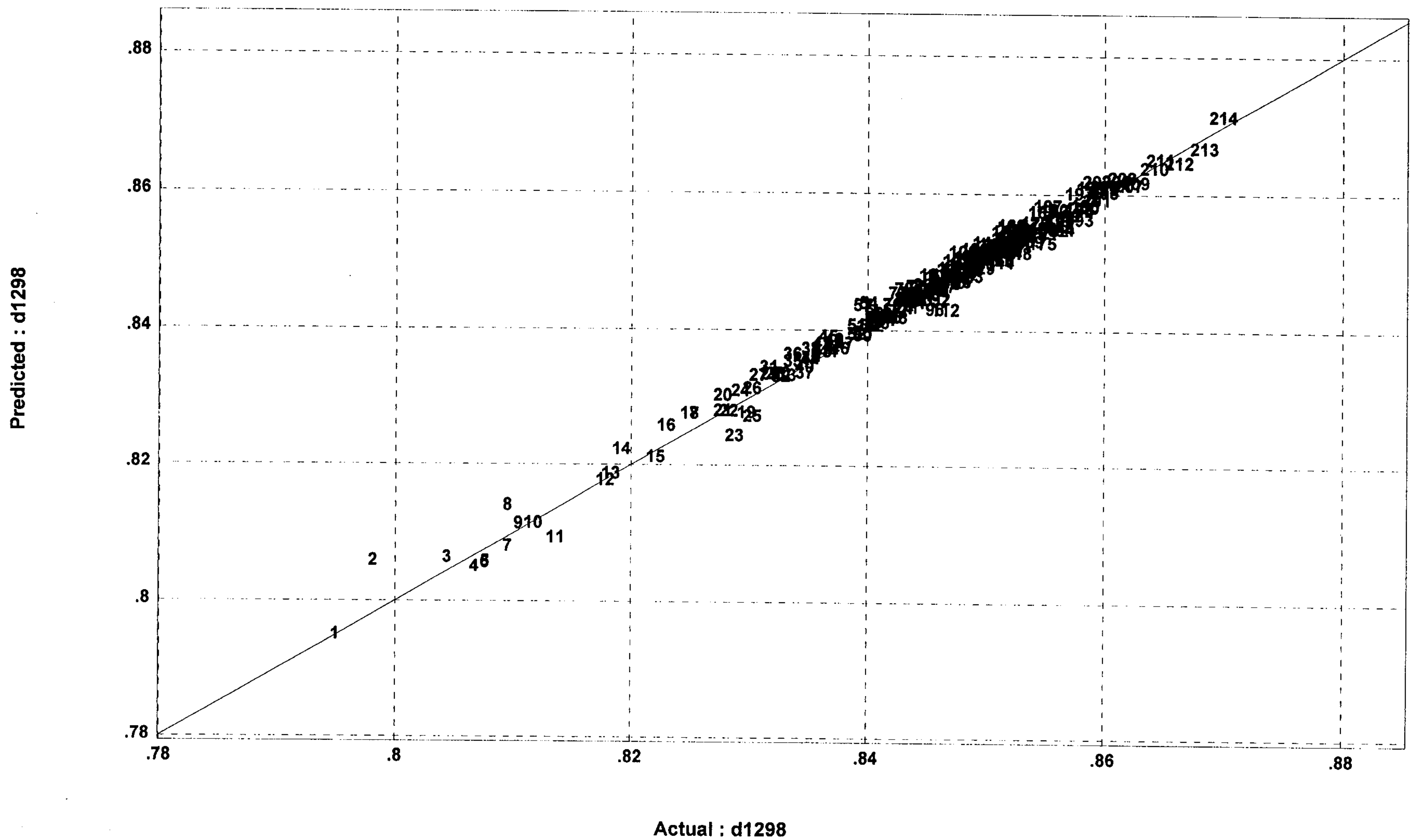
Calibration Summary: 23 components, 214 spectra, 351 points, 1 rotation sample, PLS1, mean centering

Component	Factor(recommended)	SEP(CV)	R^2
DENSITY (D 1298)	15	0.0014953	0.986615
DENSITY(D 4052)	16	0.0012824	0.990349
FLASH	3	6.0044	0.358277
CLOUD	11	4.6293	0.728989
FREEZE	11	4.9564	0.711955
POUR	11	5.8482	0.718489
VISCOSITY	18	0.12842	0.920352
BOILING PT @50%	17	4.3022	0.941754
CETANE	10	2.1716	0.581289
CARBON	3	0.27072	0.531725
HYDROGEN	7	0.091891	0.907951
CARBON/HYDROGEN	8	0.042985	0.937044
NET Ht. Comb. MJ/Kg	4	0.066309	0.83504
GUMS	1	14.52	0.000010116
WATER	11	12.648	0.267377
AROMATICS, mono-	18	0.86759	0.962601
AROMATICS, di-	10	0.54963	0.925082
AROMATICS, tri-	10	0.26714	0.726121
TOTAL AROMATICS	14	0.72578	0.985547
SULFUR	4	0.019246	0.0779117
HFRR	9	0.068022	0.580857
SLWT	6	616.02	0.241847
BOCLE	2	0.031217	0.0336625

TE: .0218233

R^2: .986615

RMSD: .00149181



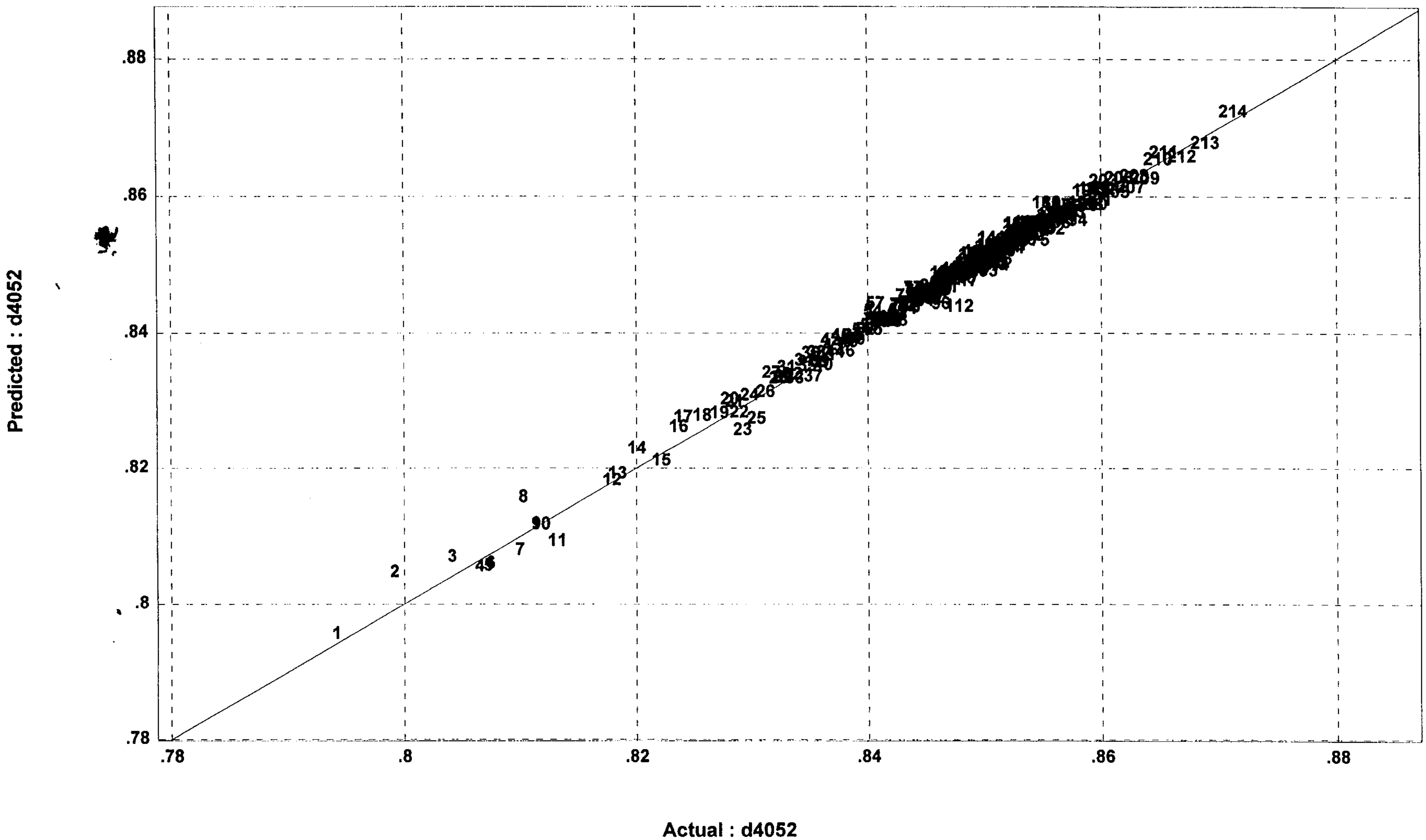
Total Factors: 15

Component: d1298

TE: .0187167

R^2: .990349

RMSD: .00127944



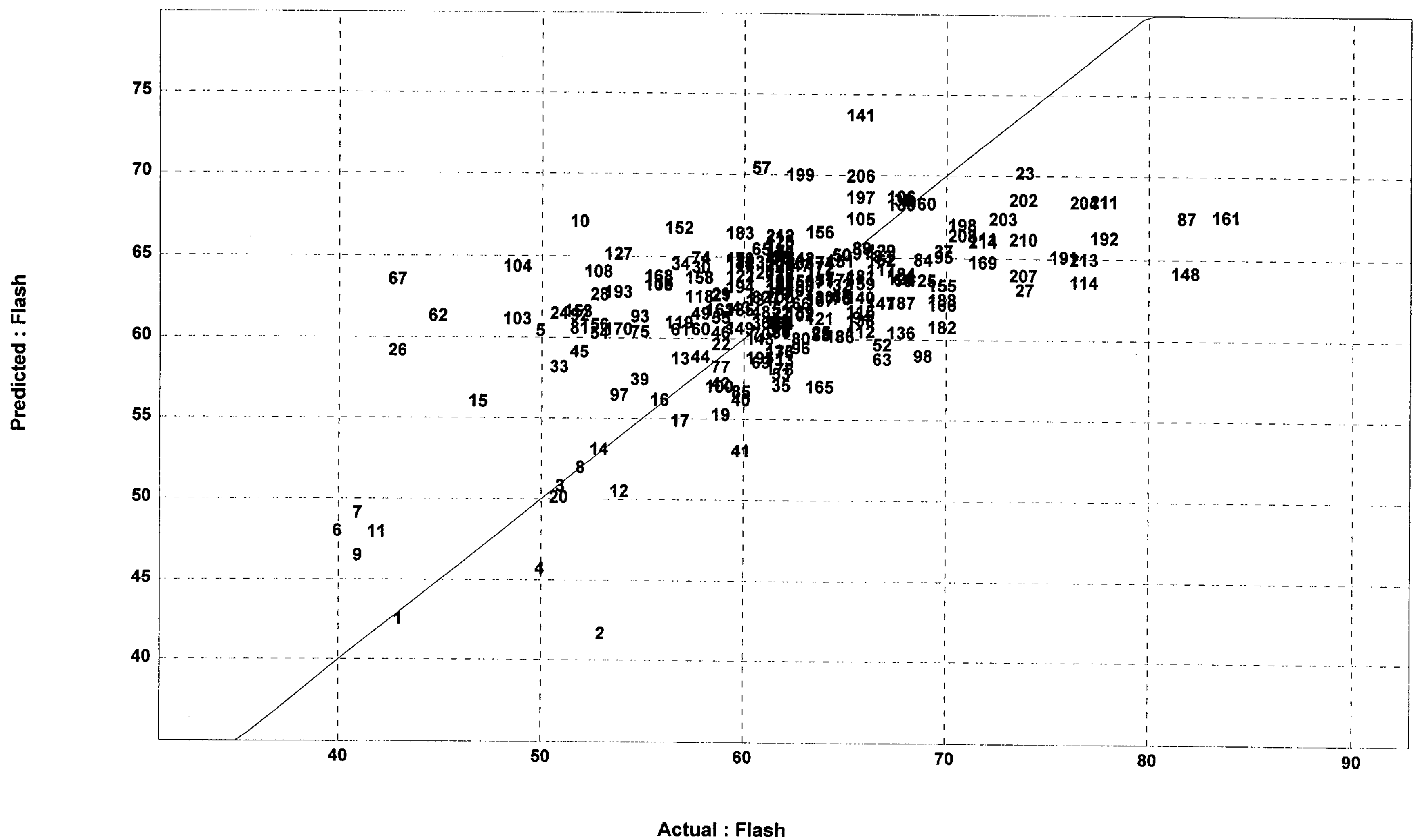
Total Factors: 16

Component: d4052

TE: 87.6312

R^2: .358277

RMSD: 5.99034



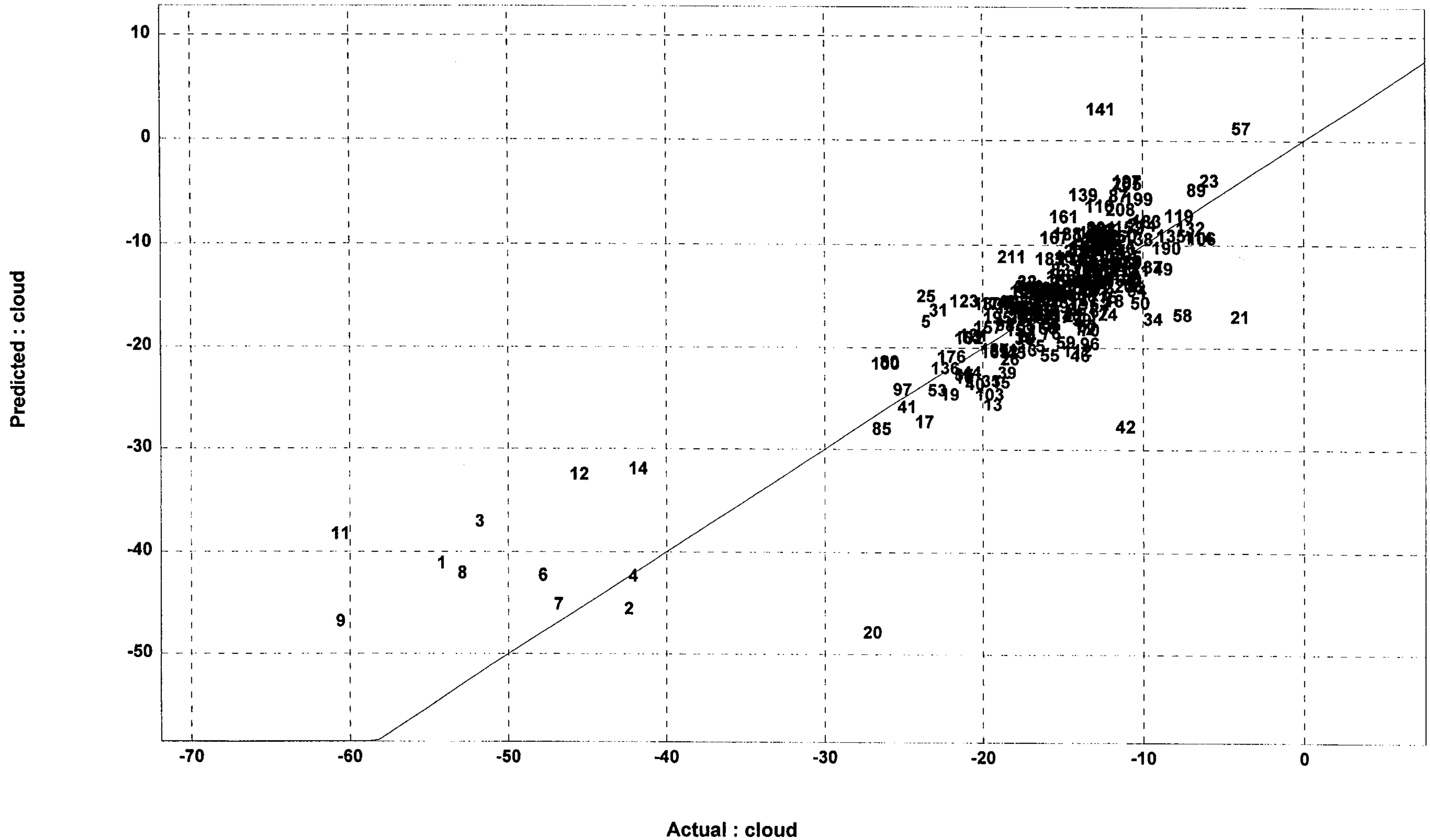
Total Factors: 3

Component: Flash

TE: 67.5627

R^2: .728989

RMSD: 4.61849



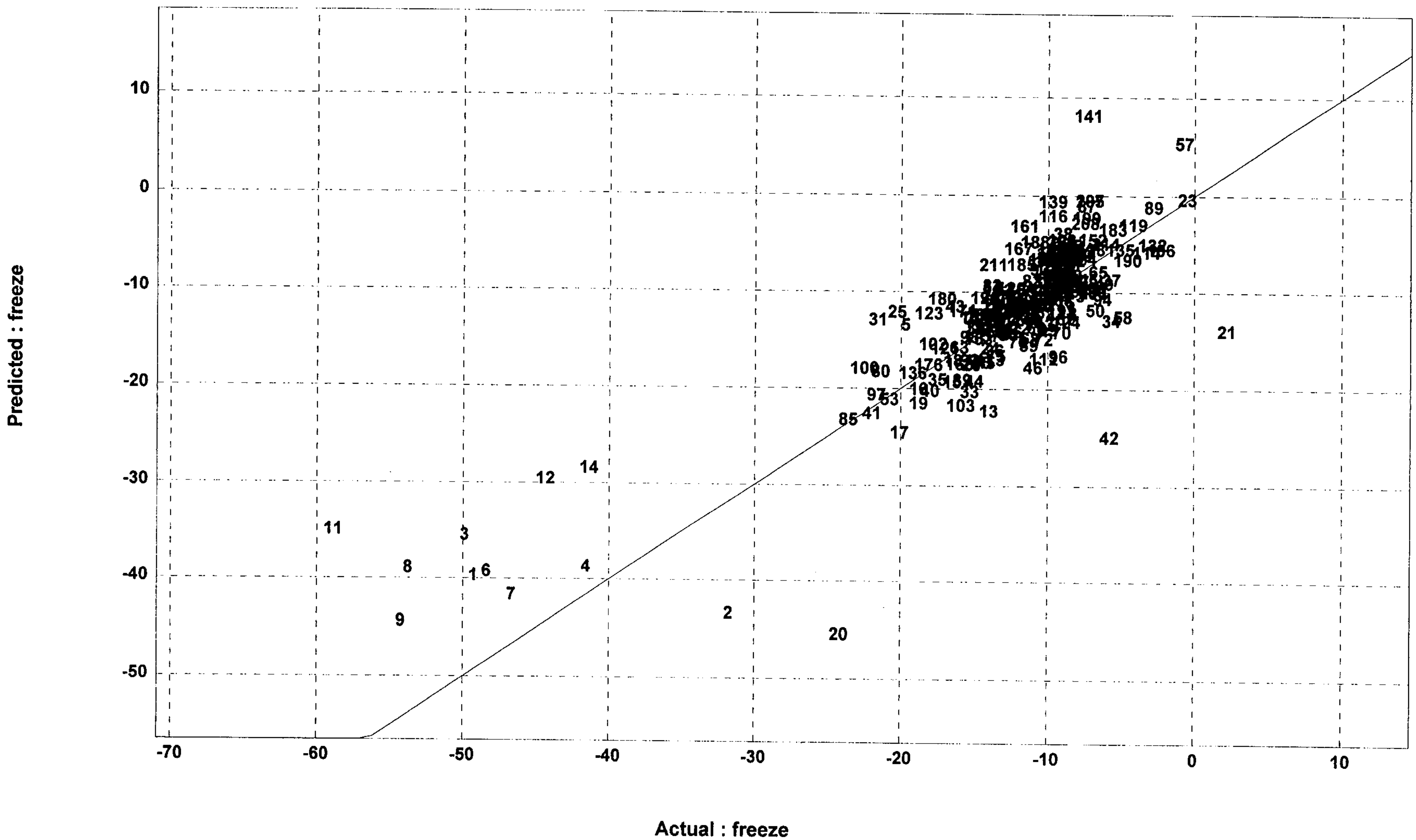
Total Factors: 11

Component: cloud

TE: 72.3363

R^2: .711955

RMSD: 4.94481



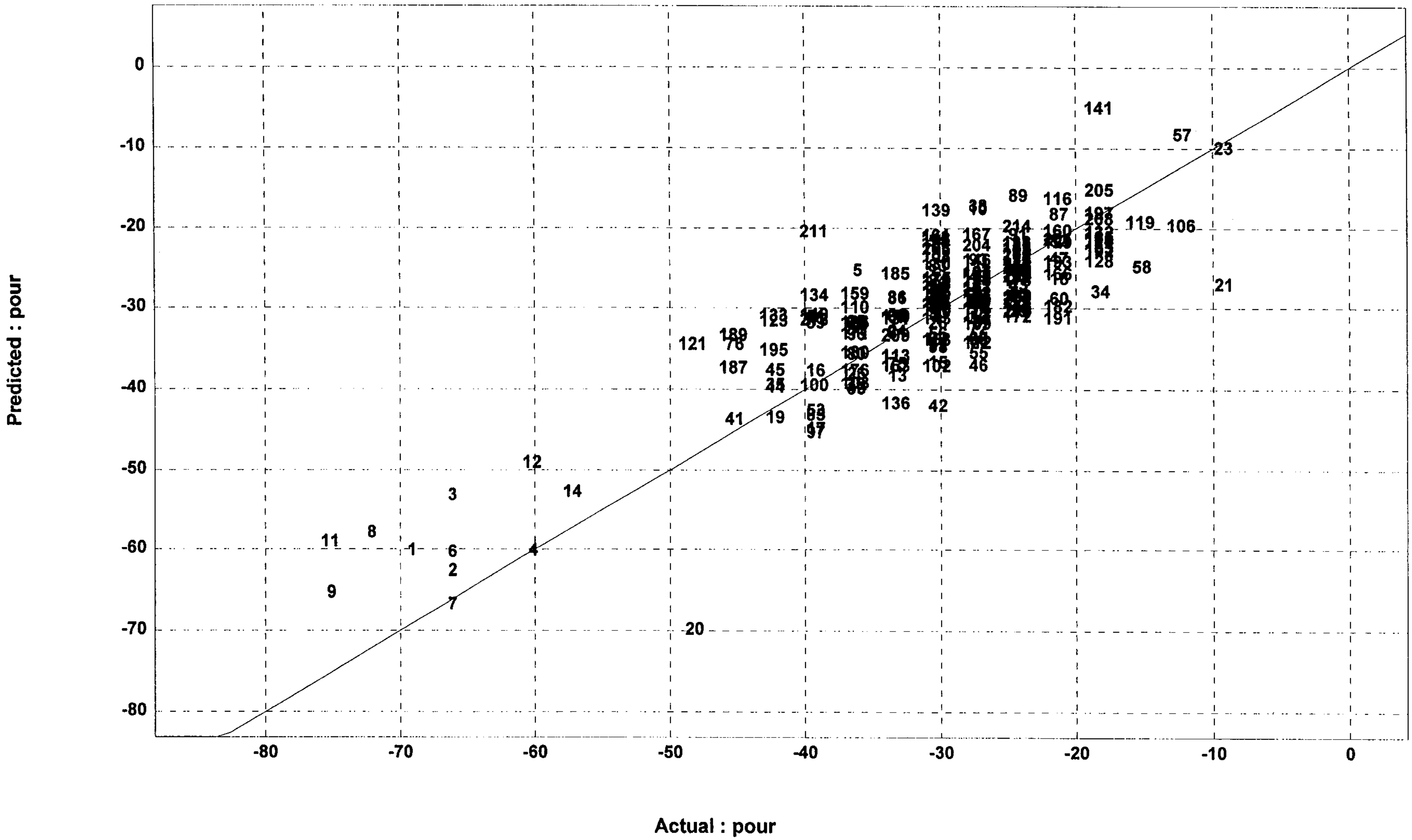
Total Factors: 11

Component: freeze

TE: 85.3525

R^2: .718489

RMSD: 5.83458



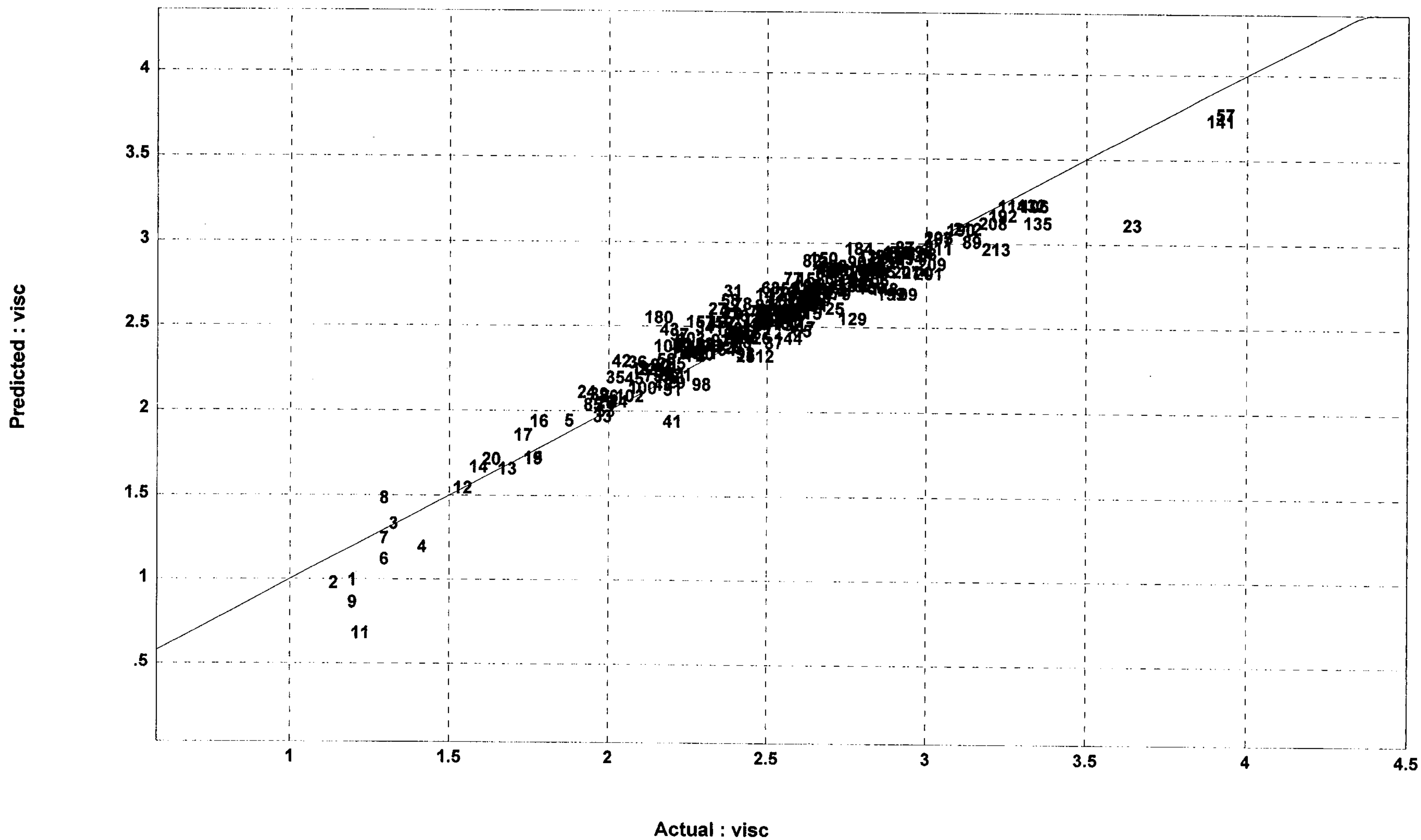
Total Factors: 11

Component: pour

TE: 1.87418

R^2: .920352

RMSD: .128117



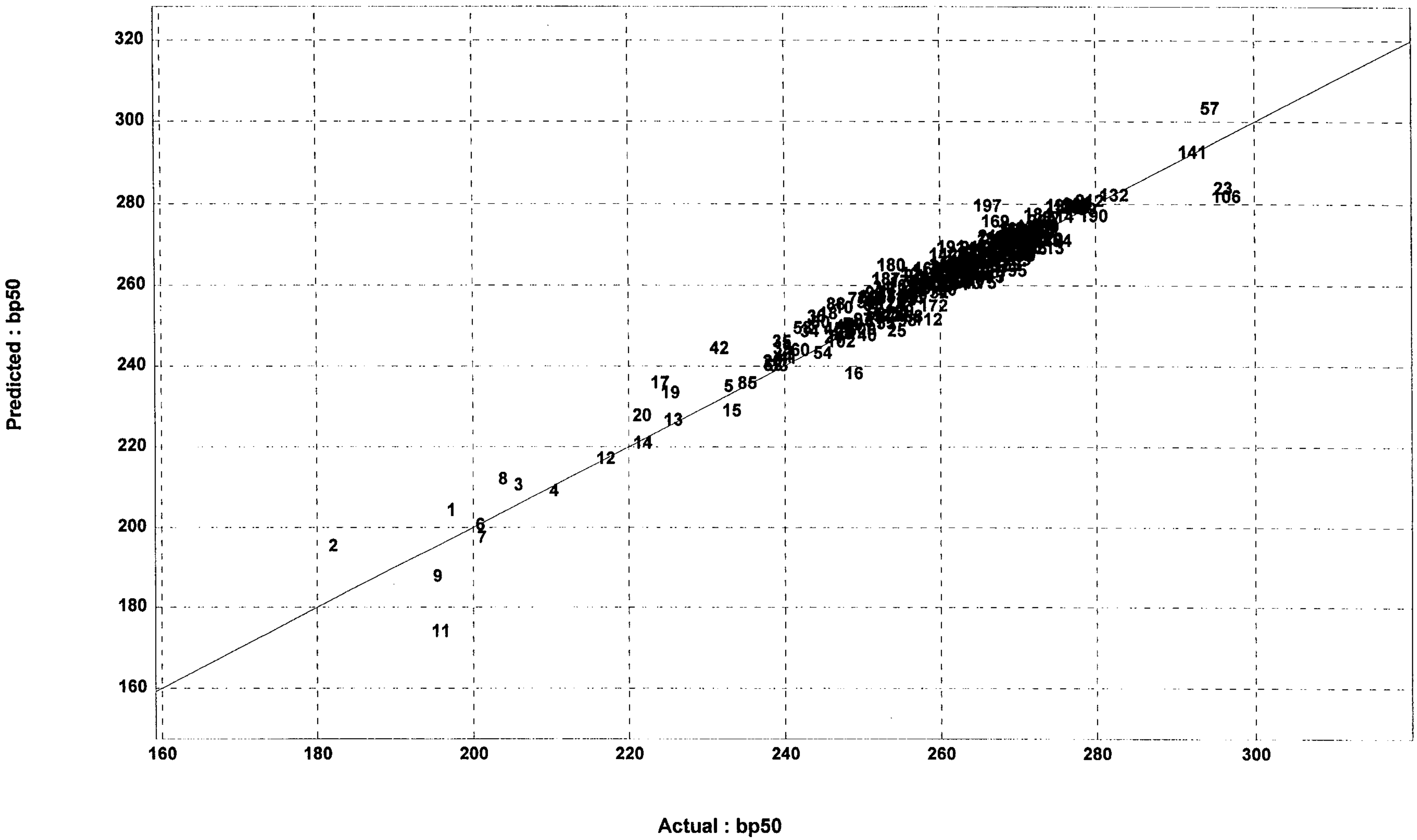
Total Factors: 18

Component: visc

TE: 62.7887

R^2: .941754

RMSD: 4.29215



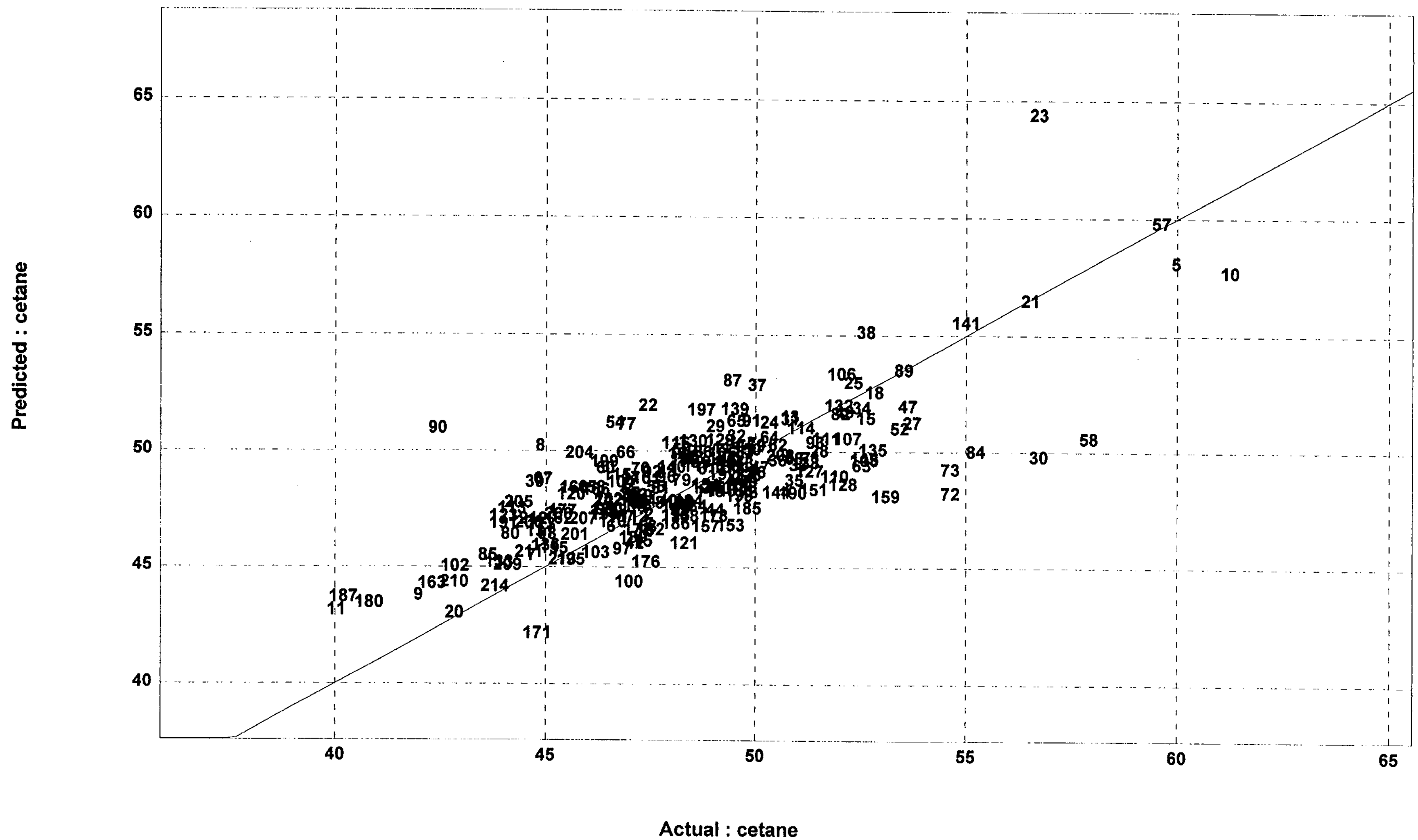
Total Factors: 17

Component: bp50

TE: 31.6935

R^2: .581289

RMSD: 2.16652



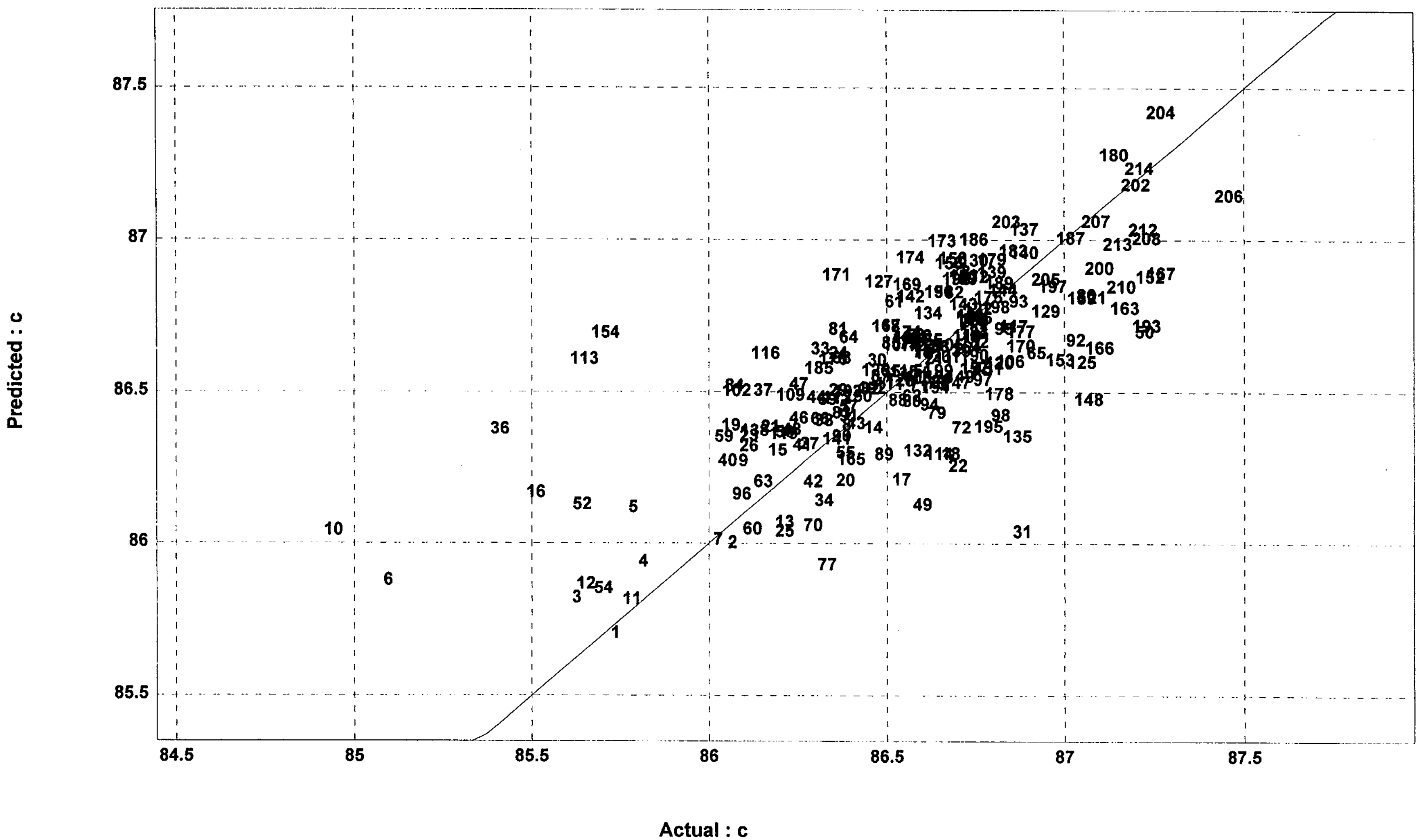
Total Factors: 10

Component: cetane

TE: 3.95109

R^2: .531725

RMSD: .270091



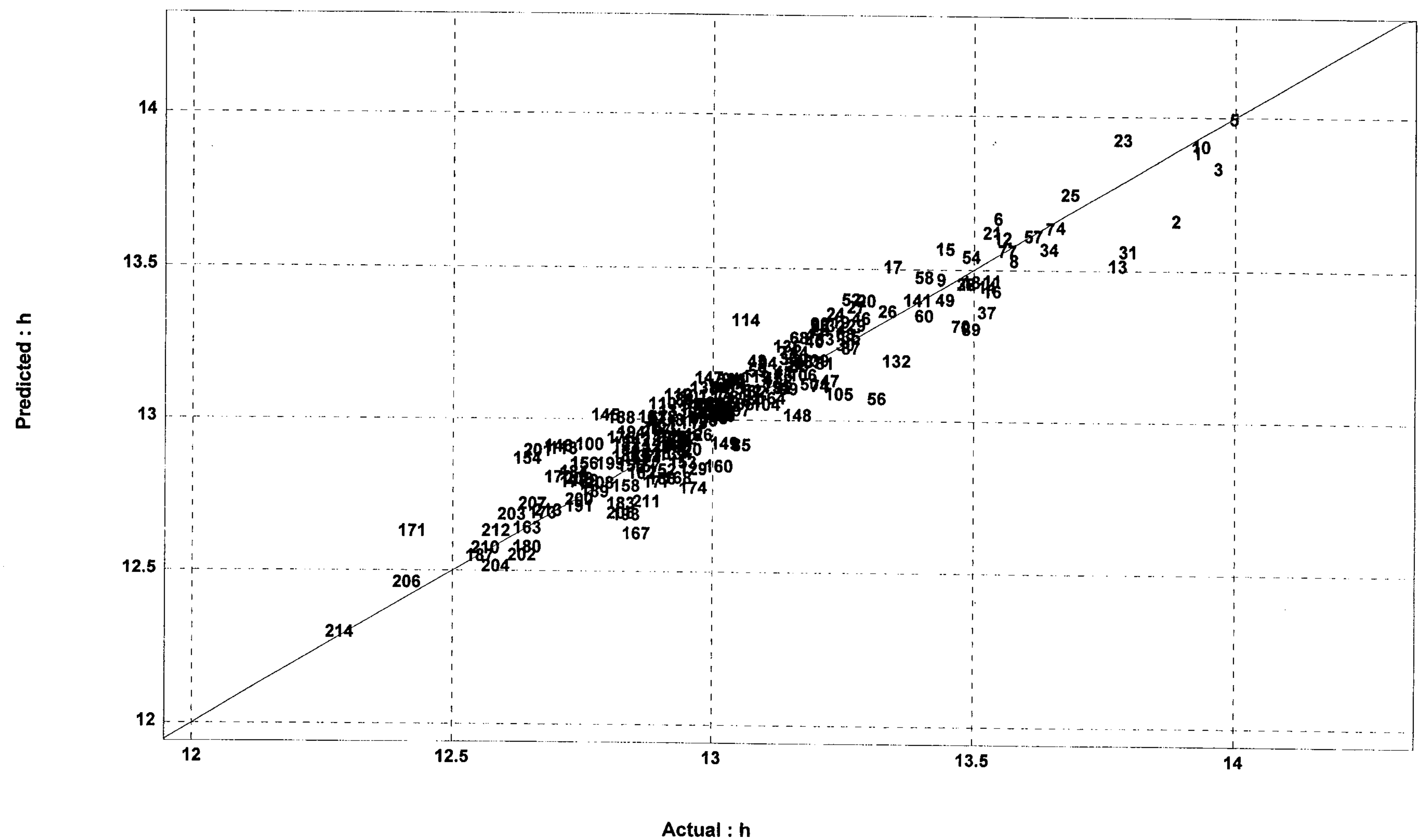
Total Factors: 3

Component: c

TE: 1.3411

R^2: .907951

RMSD: .0916758



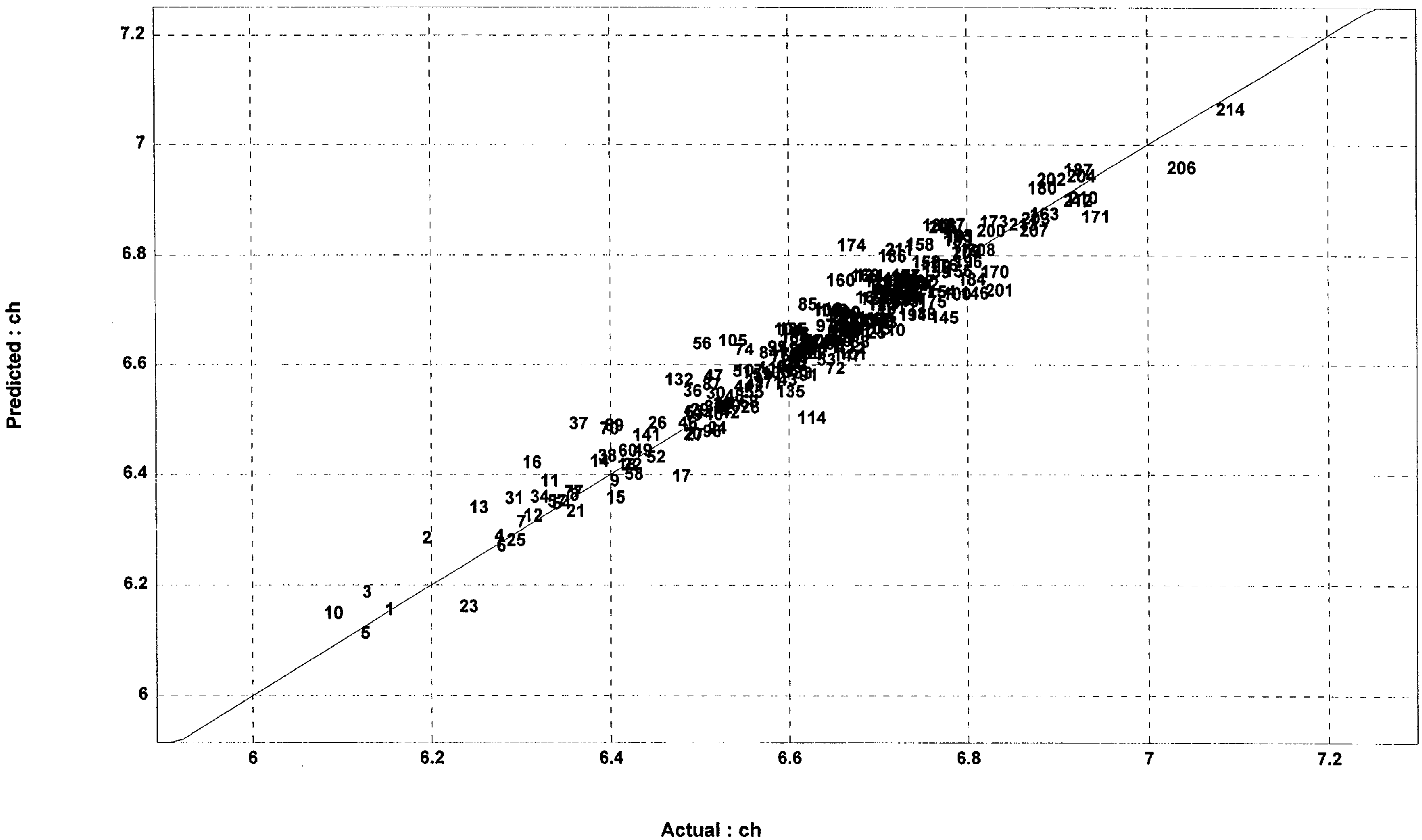
Total Factors: 7

Component: h

TE: .62735

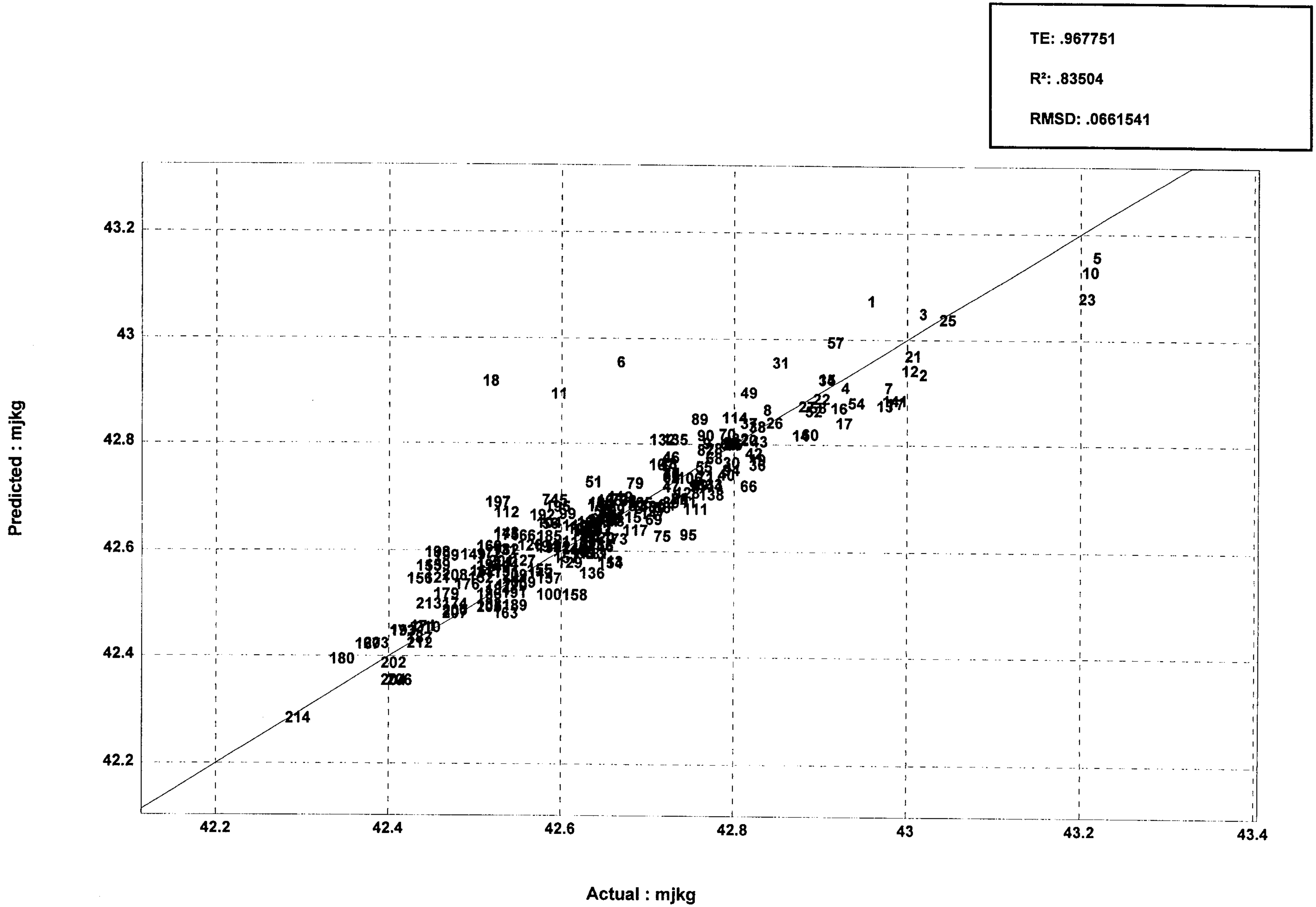
R^2: .937044

RMSD: .0428848



Total Factors: 8

Component: ch



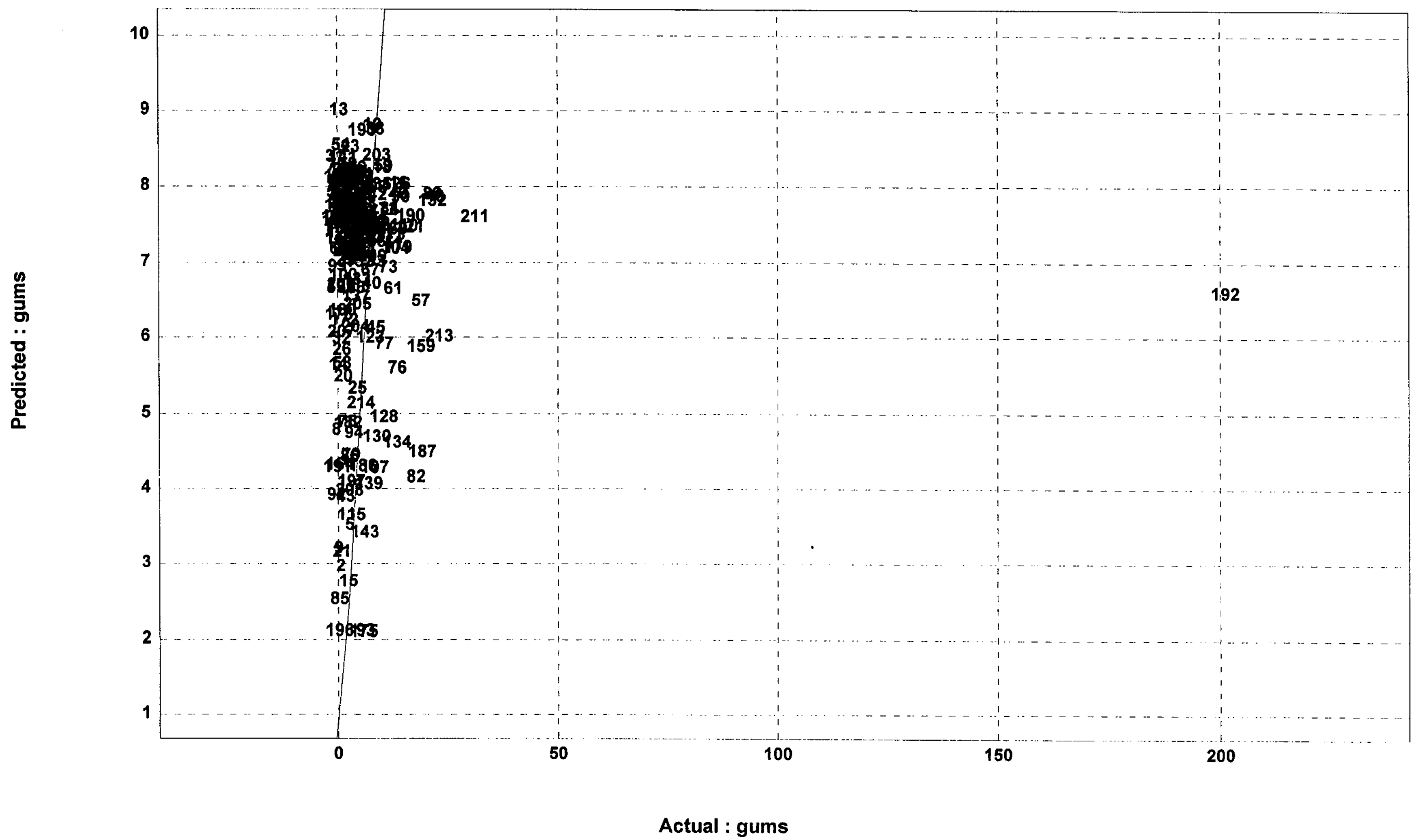
Total Factors: 4

Component: mjkg

TE: 211.913

R^2: .000010116

RMSD: 14.4861



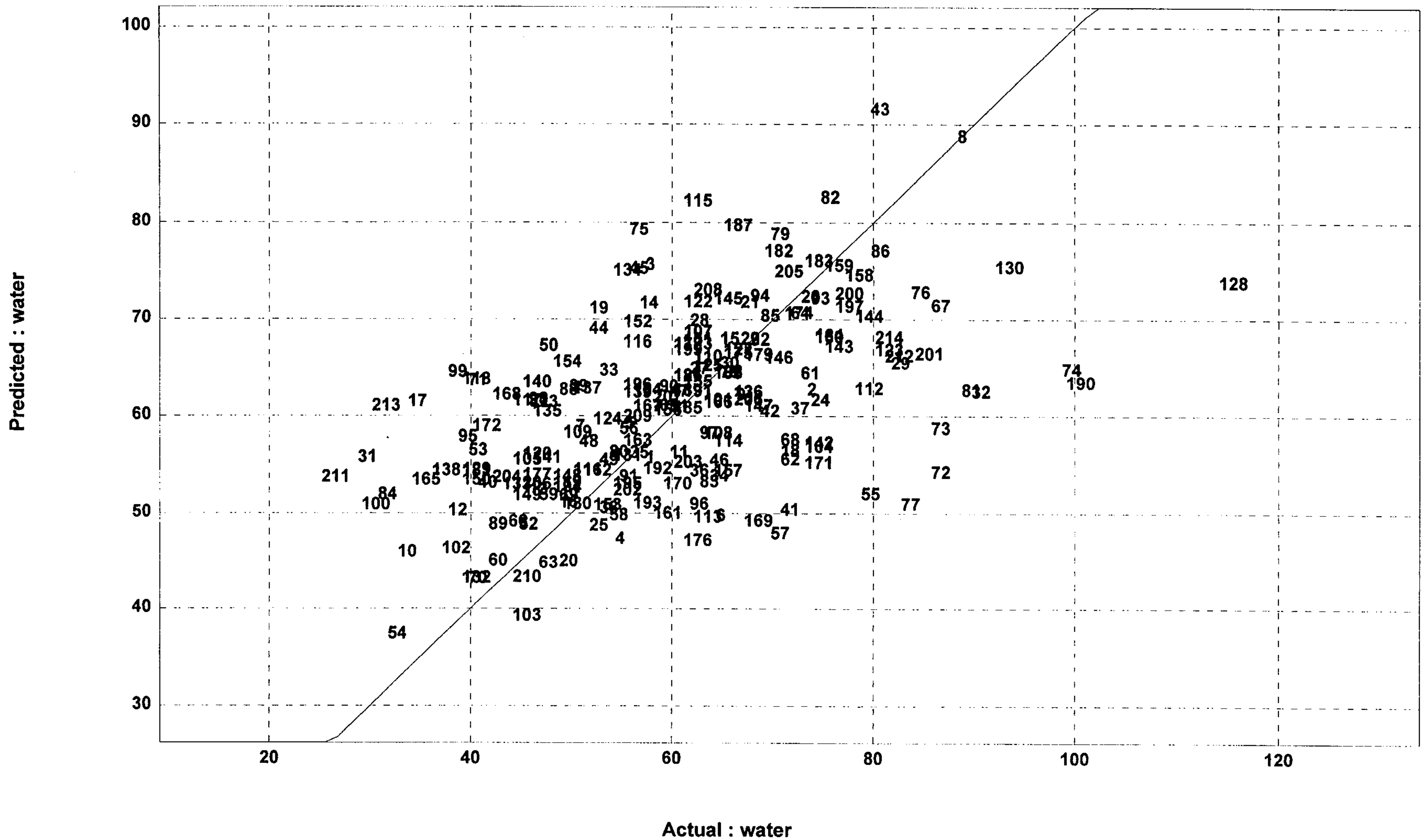
Total Factors: 1

Component: gums

TE: 184.59

R^2: .267377

RMSD: 12.6183



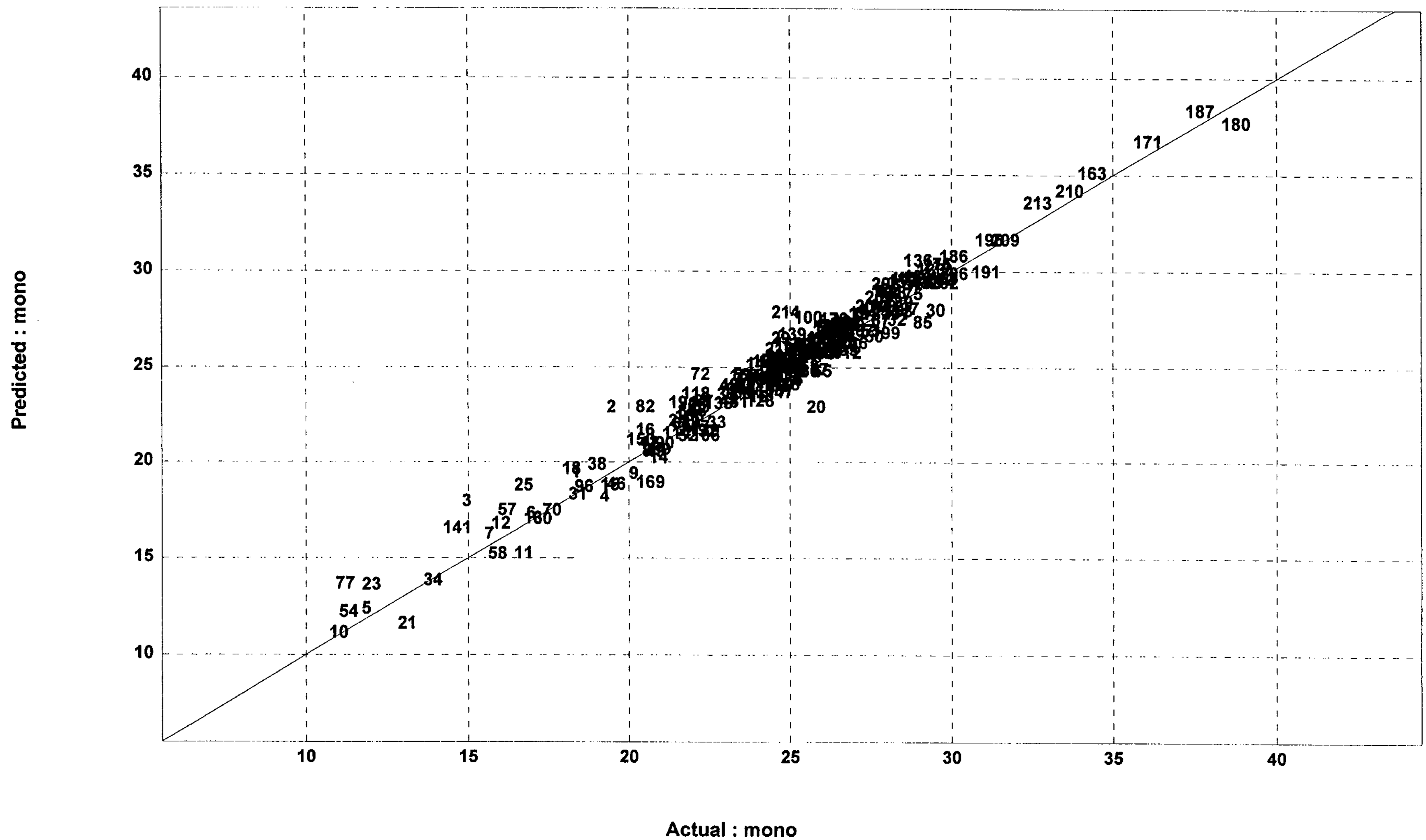
Total Factors: 11

Component: water

TE: 12.662

R^2: .962601

RMSD: .865559



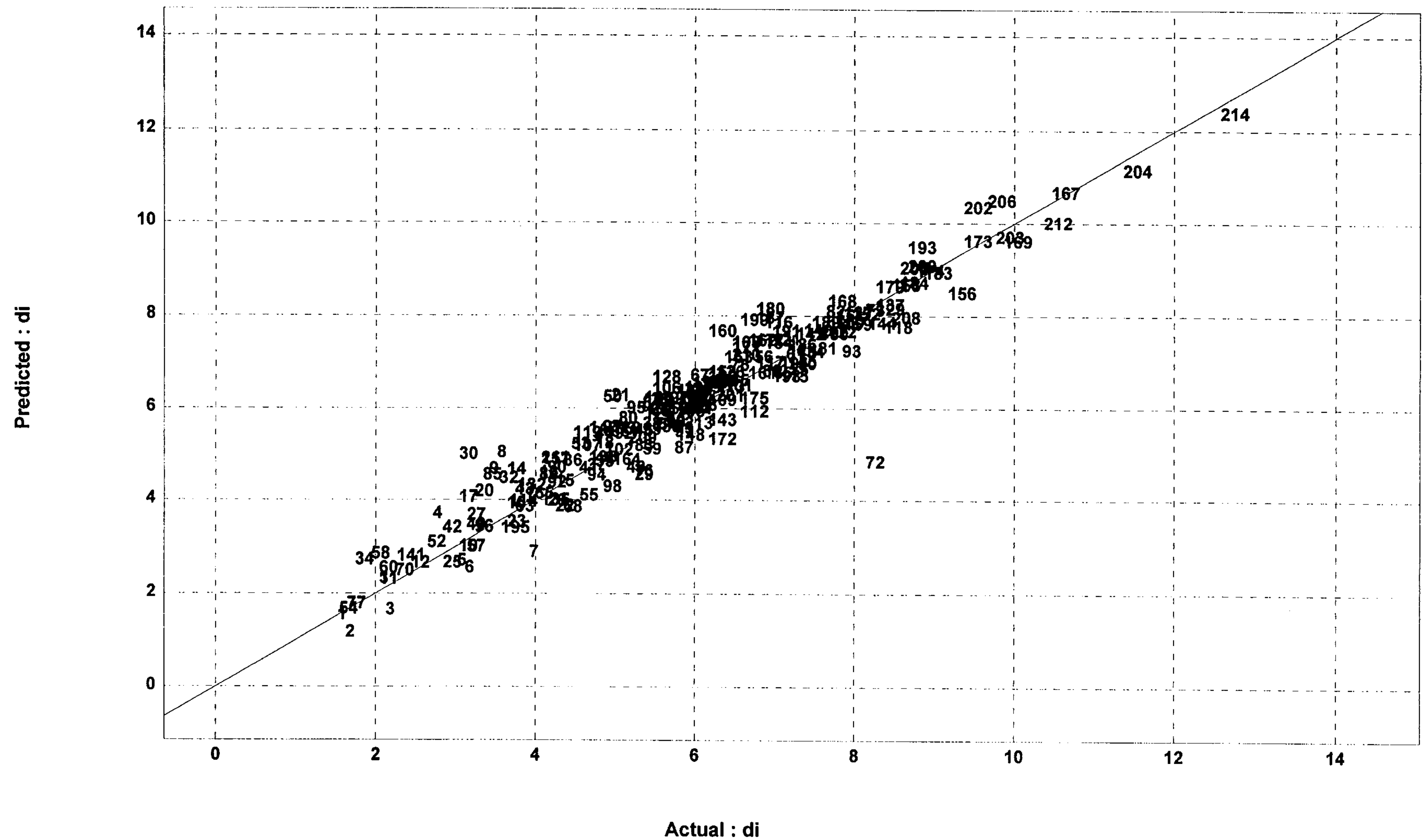
Total Factors: 18

Component: mono

TE: 8.02158

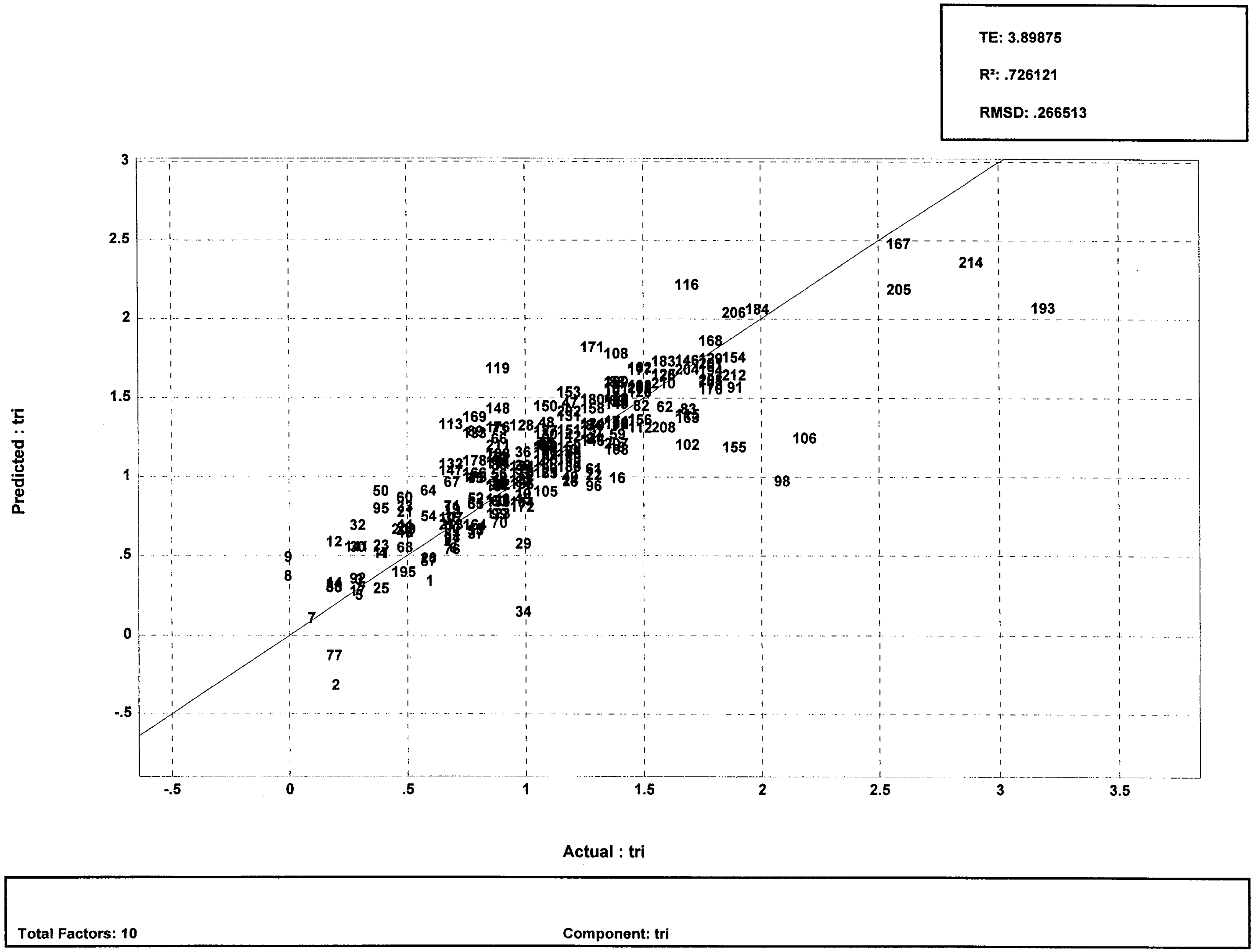
R^2: .925082

RMSD: .548344



Total Factors: 10

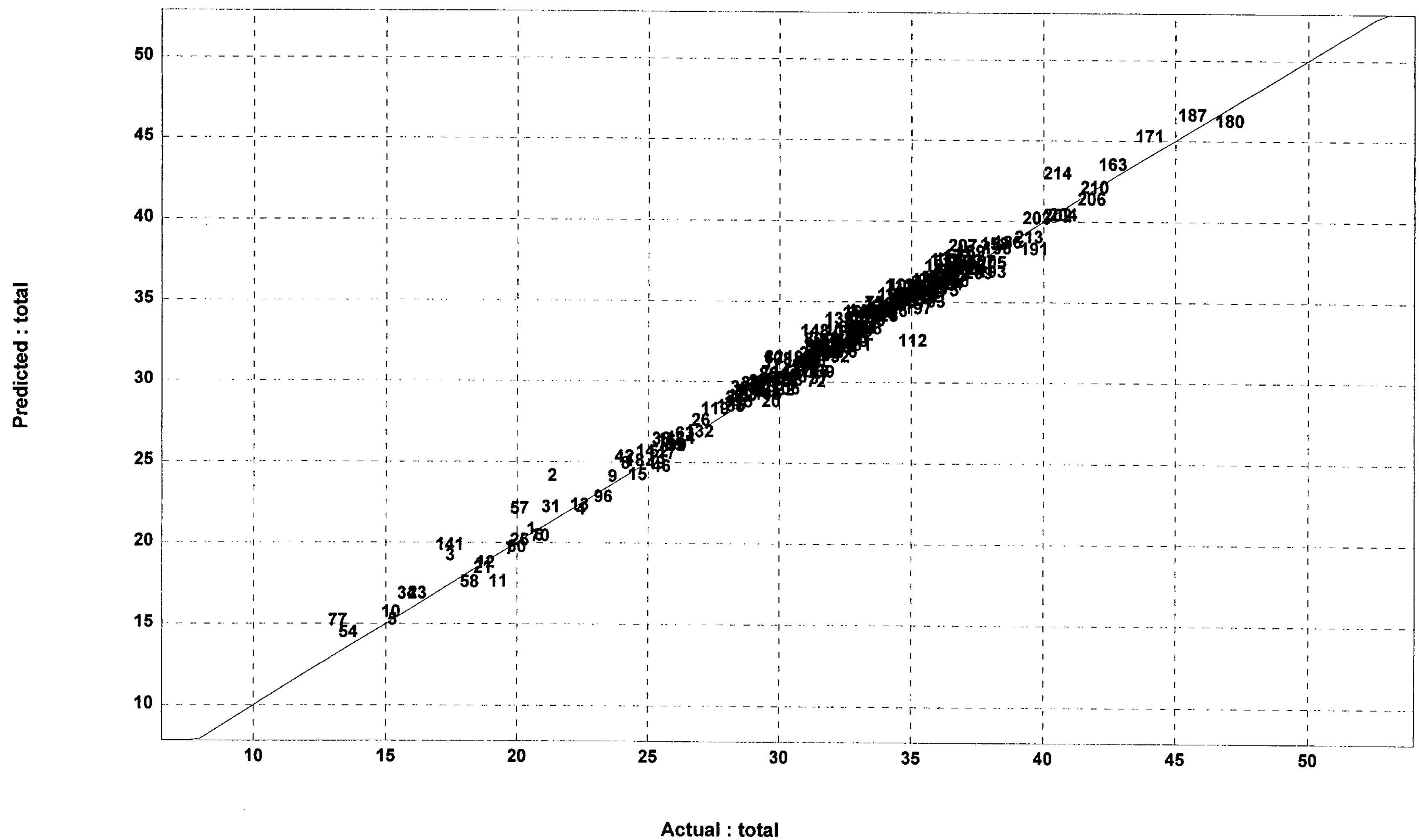
Component: di


TE: 3.89875
R²: .726121
RMSD: .266513
Predicted : tri
Actual : tri
Total Factors: 10
Component: tri

TE: 10.5924

R^2: .985547

RMSD: .724079



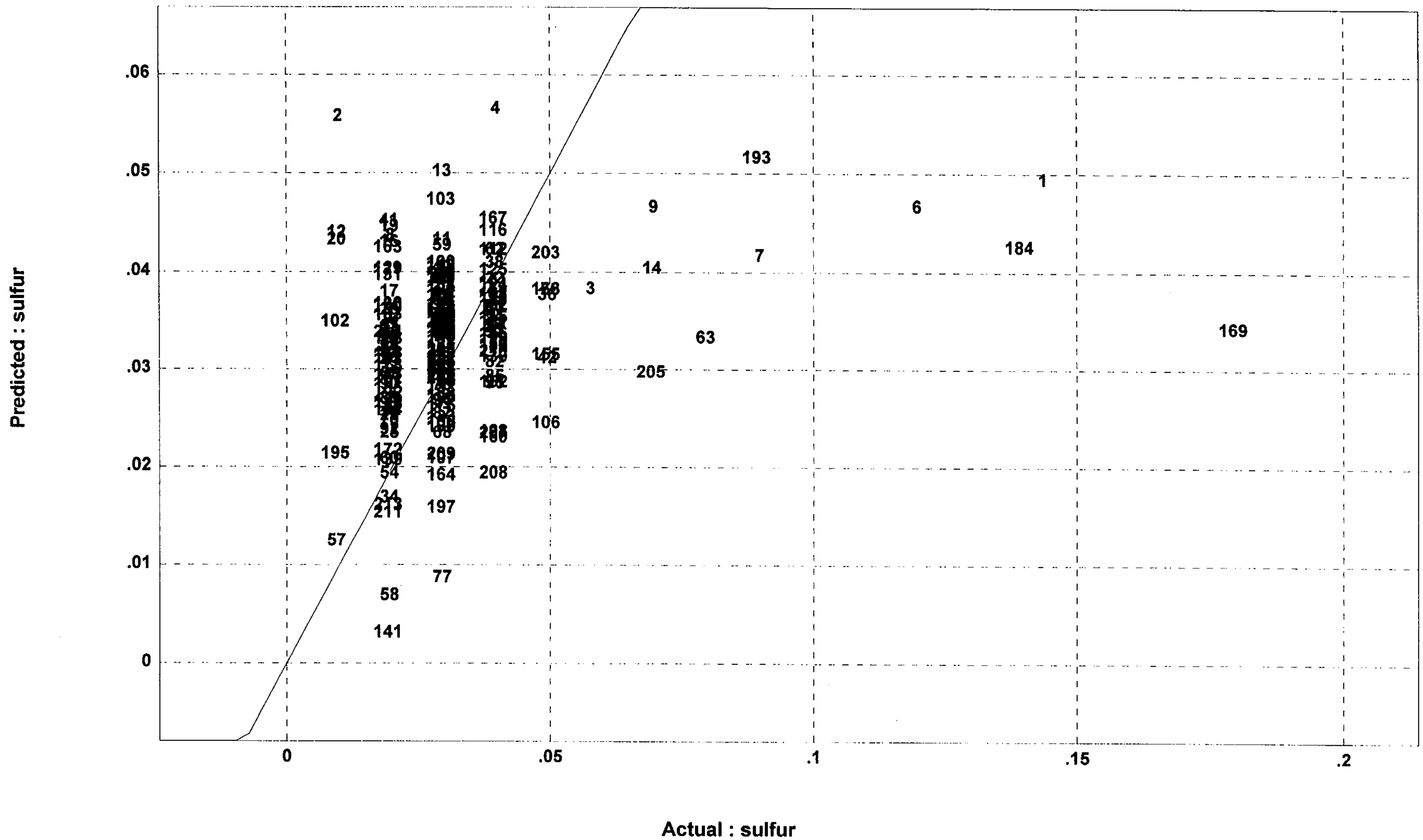
Total Factors: 14

Component: total

TE: .280883

R^2: .0779117

RMSD: .0192008



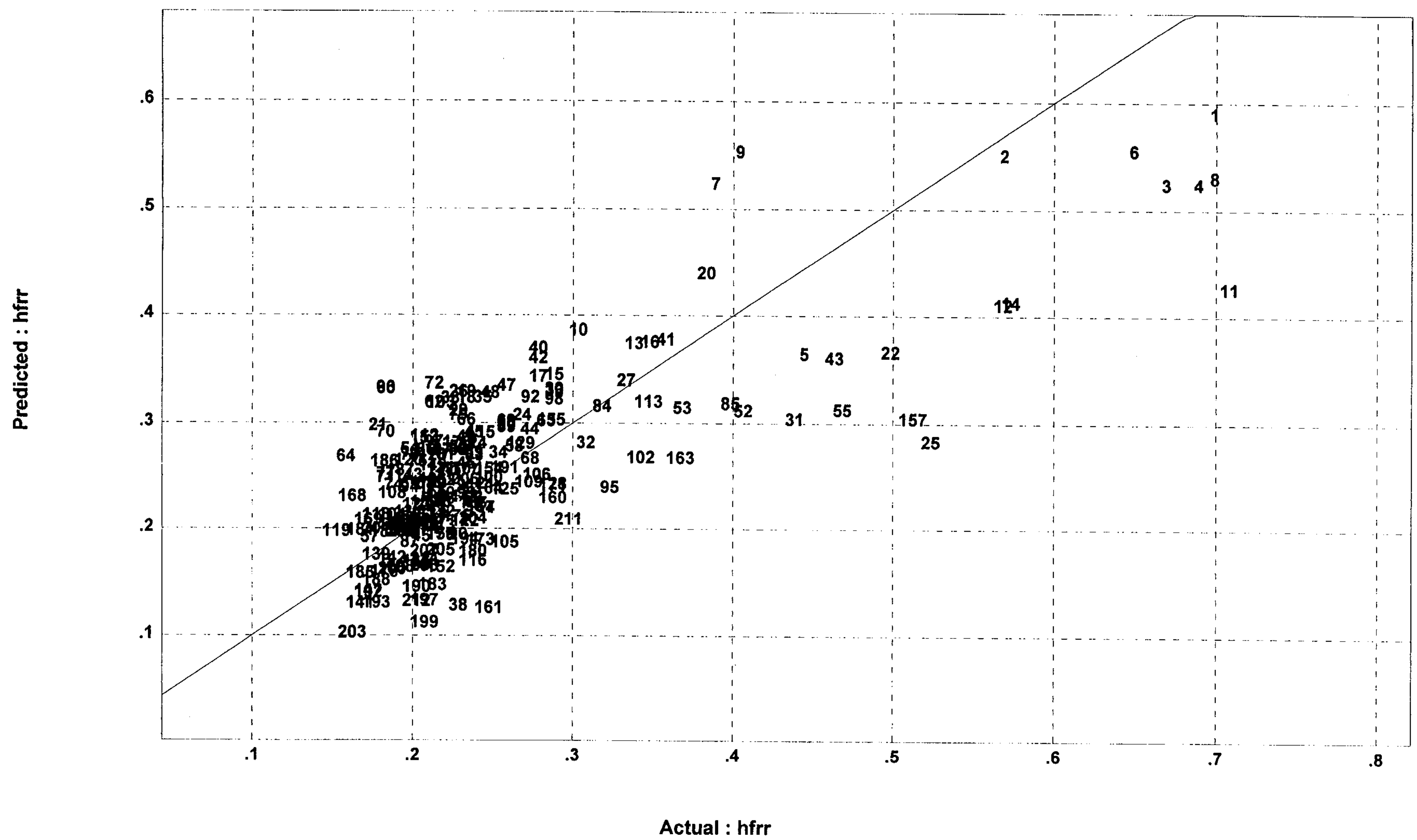
Total Factors: 4

Component: sulfur

TE: .992749

R^2: .580857

RMSD: .0678629



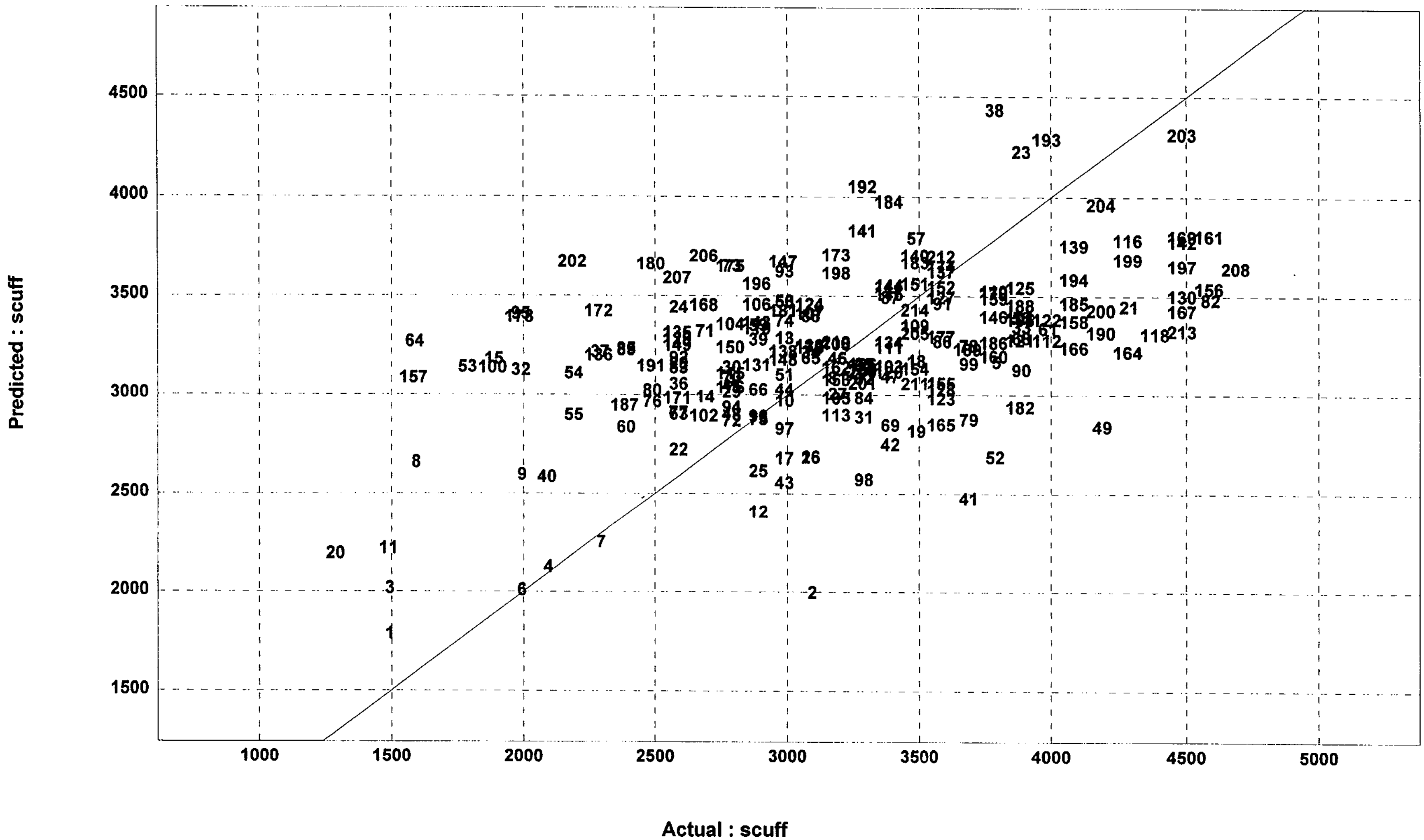
Total Factors: 9

Component: hfrr

TE: 8990.54

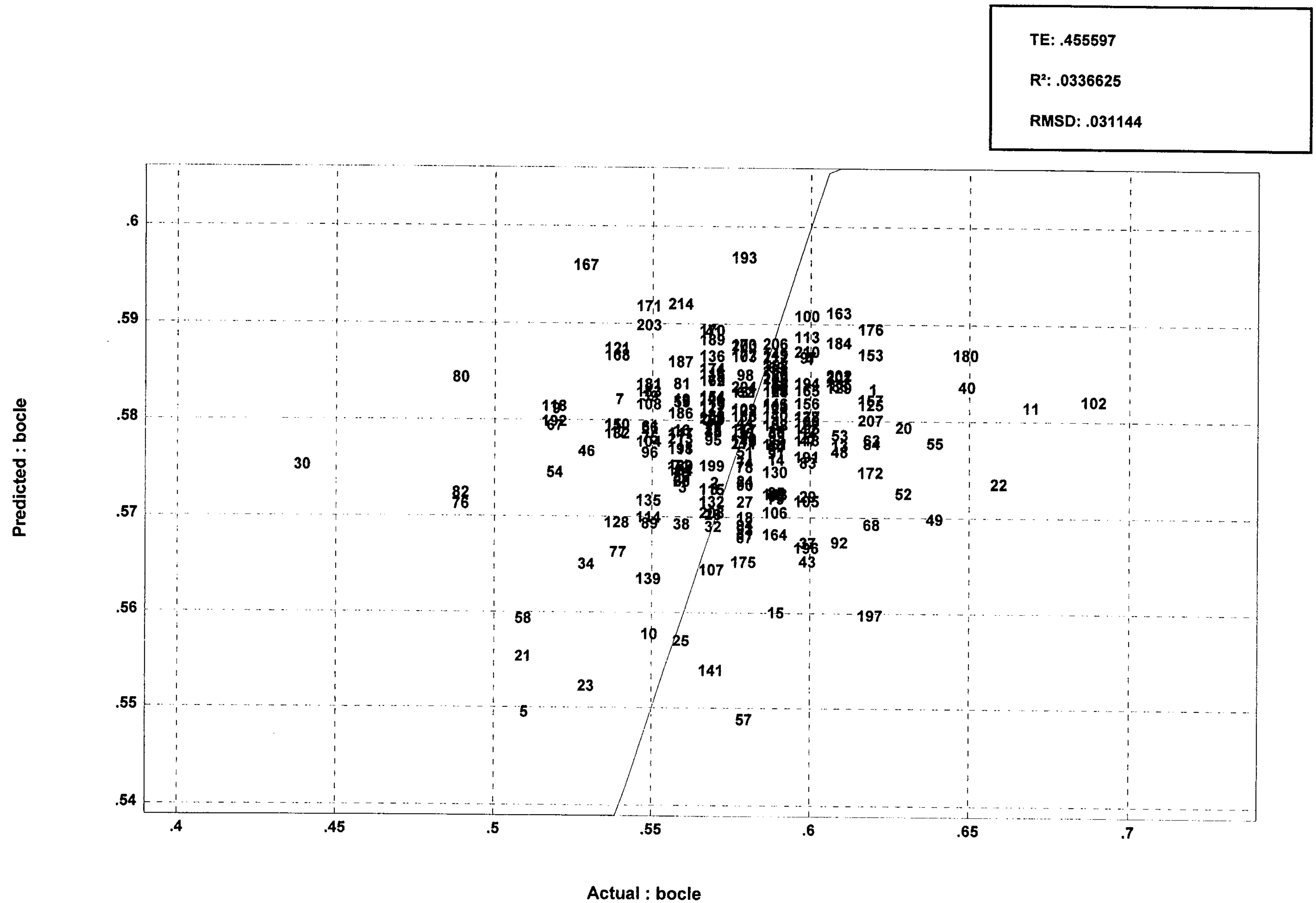
R^2: .241847

RMSD: 614.58



Total Factors: 6

Component: scuff



Total Factors: 2

Component: bocle

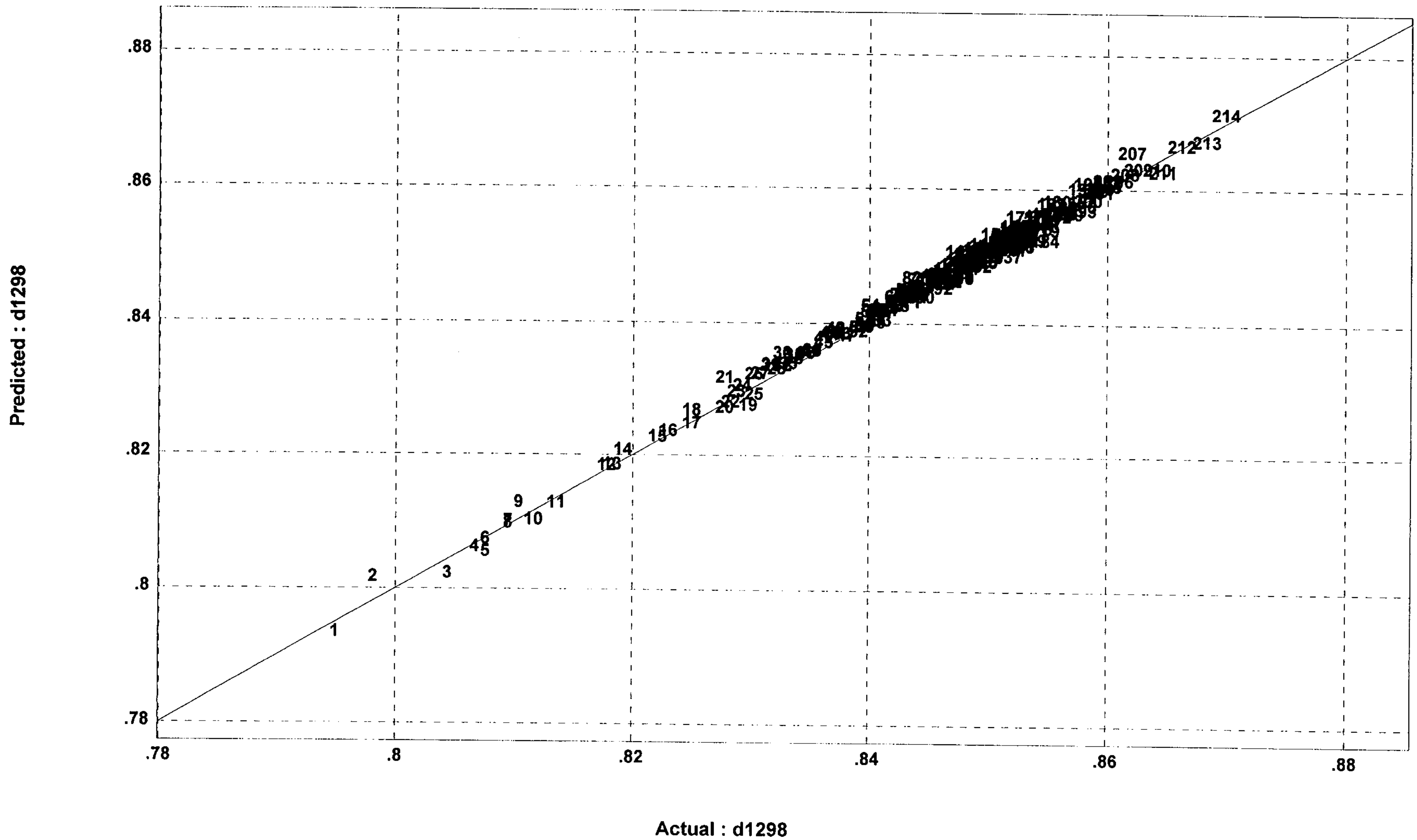
Instrument: Infrared Fiber Systems(IFS)
Calibration Summary: 23 components, 214 spectra, 600 points, 1 rotation sample, PLS1, mean centering

Component	Factor(recommended)	SEP(CV)	R^2
DENSITY (D 1298)	17	0.0011091	0.99266
DENSITY(D 4052)	19	0.00085204	0.99575
FLASH	5	5.85	0.39149
CLOUD	12	4.5187	0.74104
FREEZE	11	4.8941	0.71792
POUR	11	6.1248	0.6907
VISCOSITY	19	0.14993	0.89274
BOILING PT @50%	17	4.3547	0.94078
CETANE	7	2.2337	0.55461
CARBON	4	0.27784	0.50718
HYDROGEN	8	0.090071	0.91154
CARBON/HYDROGEN	9	0.040927	0.94291
NET Ht. Comb. MJ/Kg	7	0.067131	0.83102
GUMS	1	14.501	0.00075542
WATER	1	14.39	0.038936
AROMATICS, mono-	17	0.60914	0.98156
AROMATICS, di-	16	0.46653	0.94615
AROMATICS, tri-	13	0.28669	0.68649
TOTAL AROMATICS	19	0.54743	0.99178
SULFUR	14	0.018429	0.19924
HFRR	8	0.069555	0.56146
SLWT	6	633.18	0.20243
BOCLE	1	0.03172	0.0036392

TE: .0161876

R^2: .992659

RMSD: .00110656



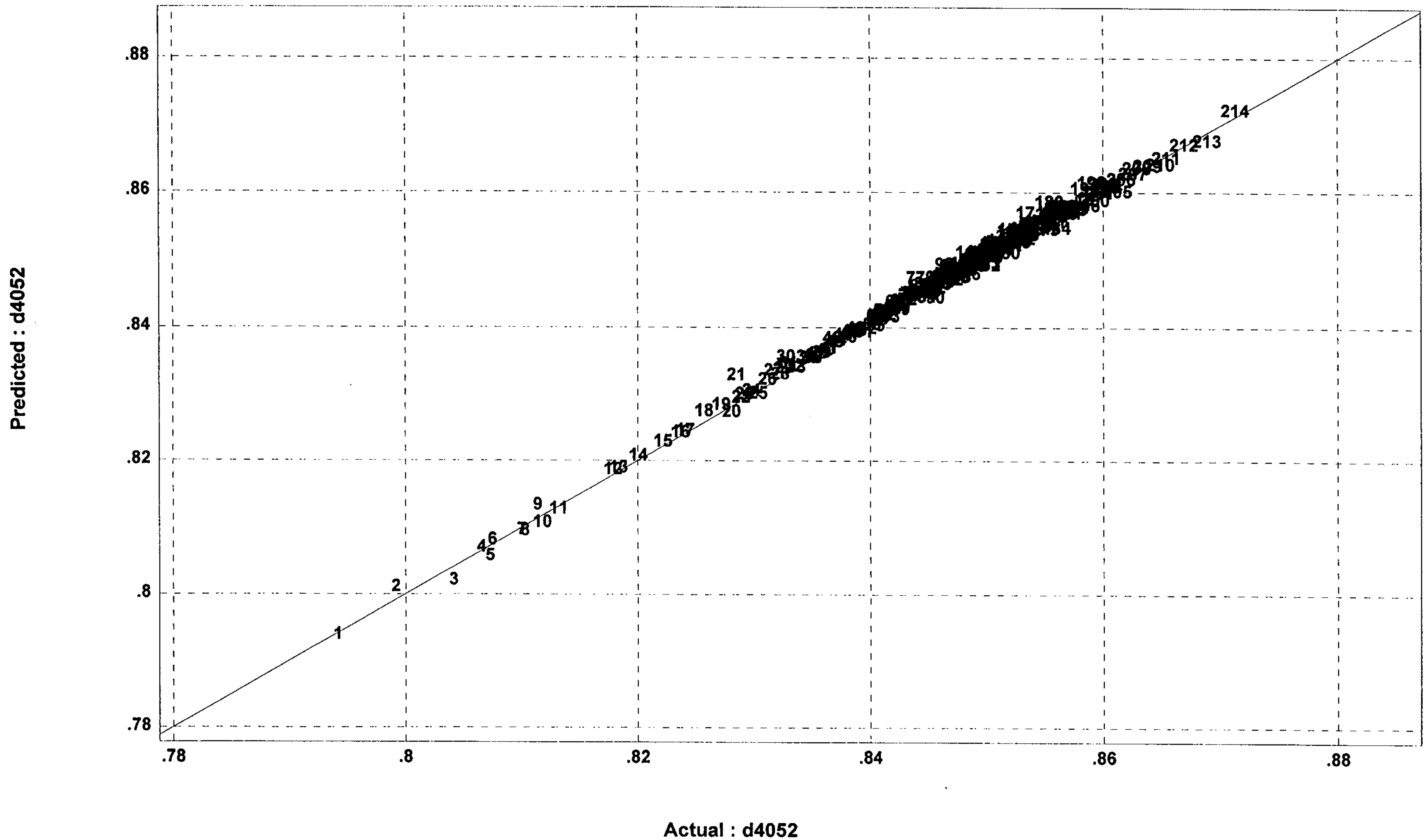
Total Factors: 17

Component: d1298

TE: .0124352

R^2: .995747

RMSD: .000850051



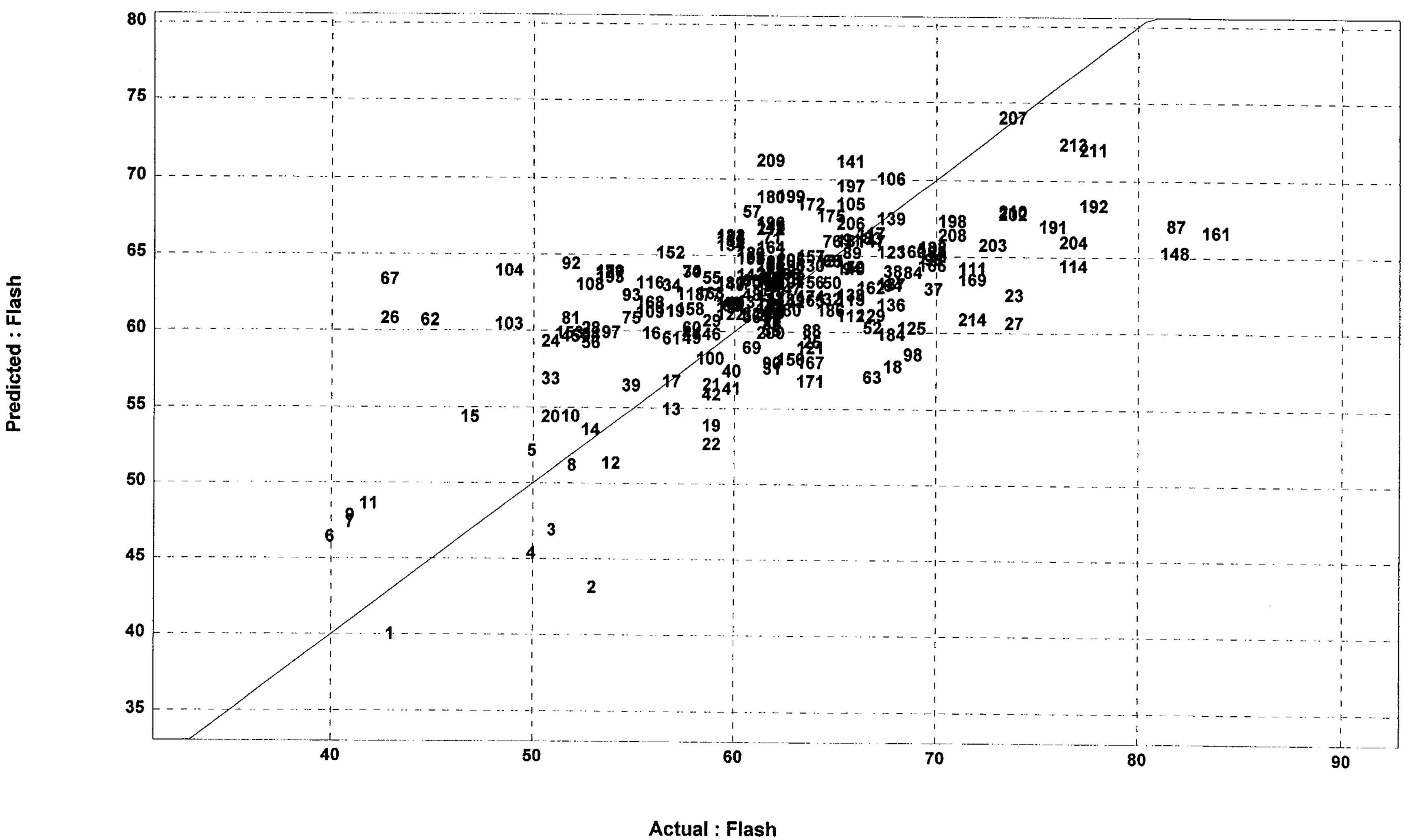
Total Factors: 19

Component: d4052

TE: 85.3783

R^2: .39149

RMSD: 5.83634



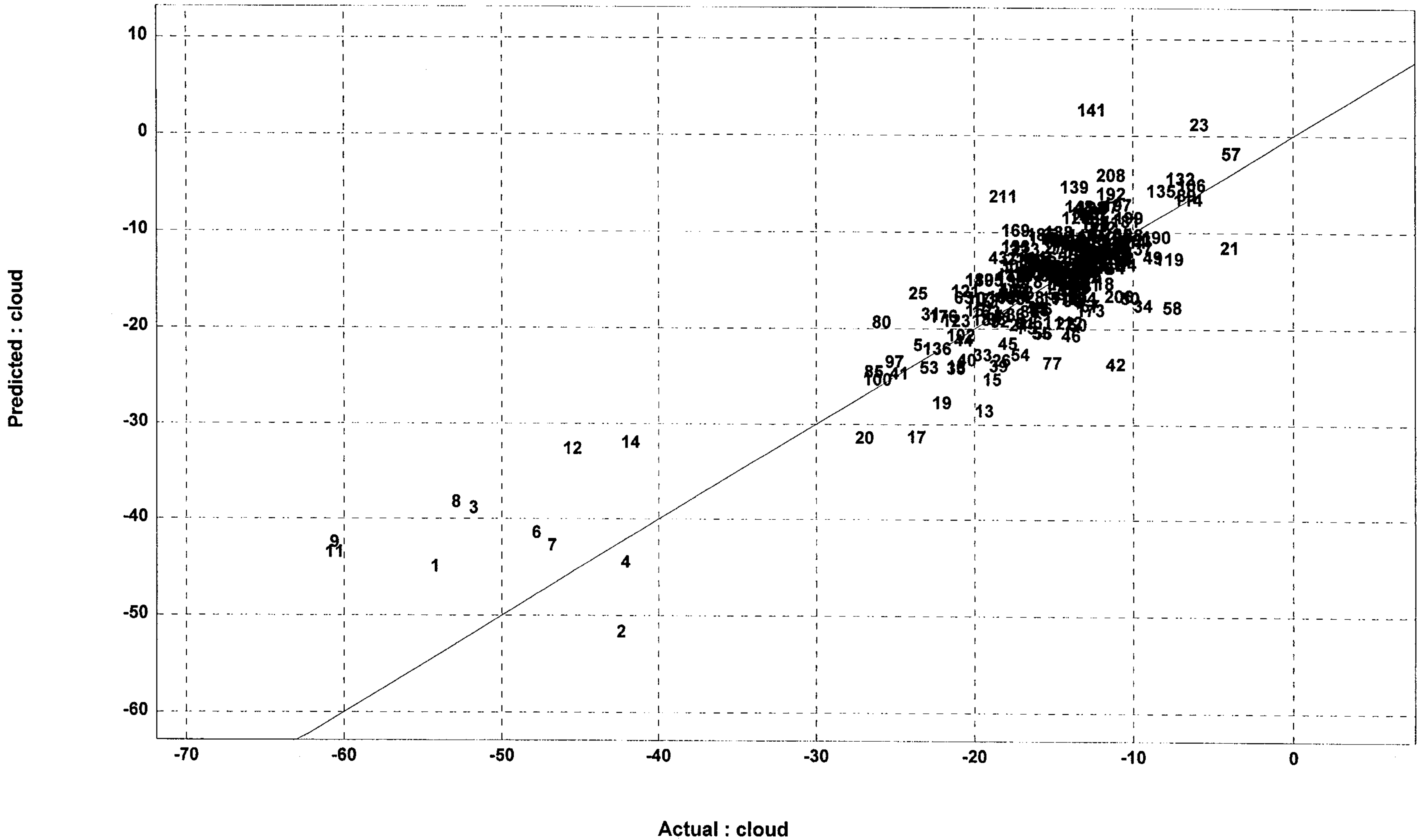
Total Factors: 5

Component: Flash

TE: 65.9479

R^2: .741044

RMSD: 4.50811



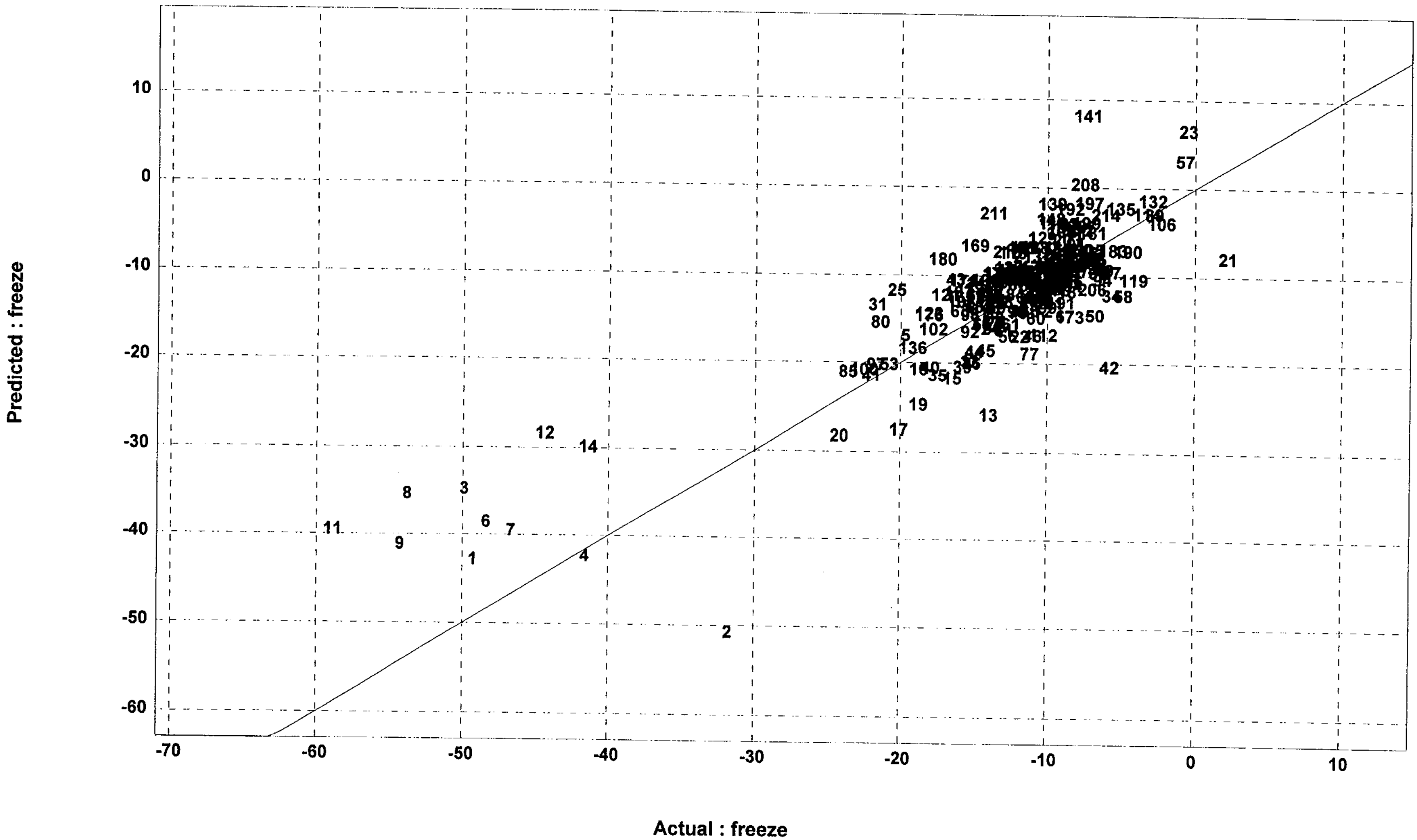
Total Factors: 12

Component: cloud

TE: 71.4268

R^2: .717918

RMSD: 4.88263



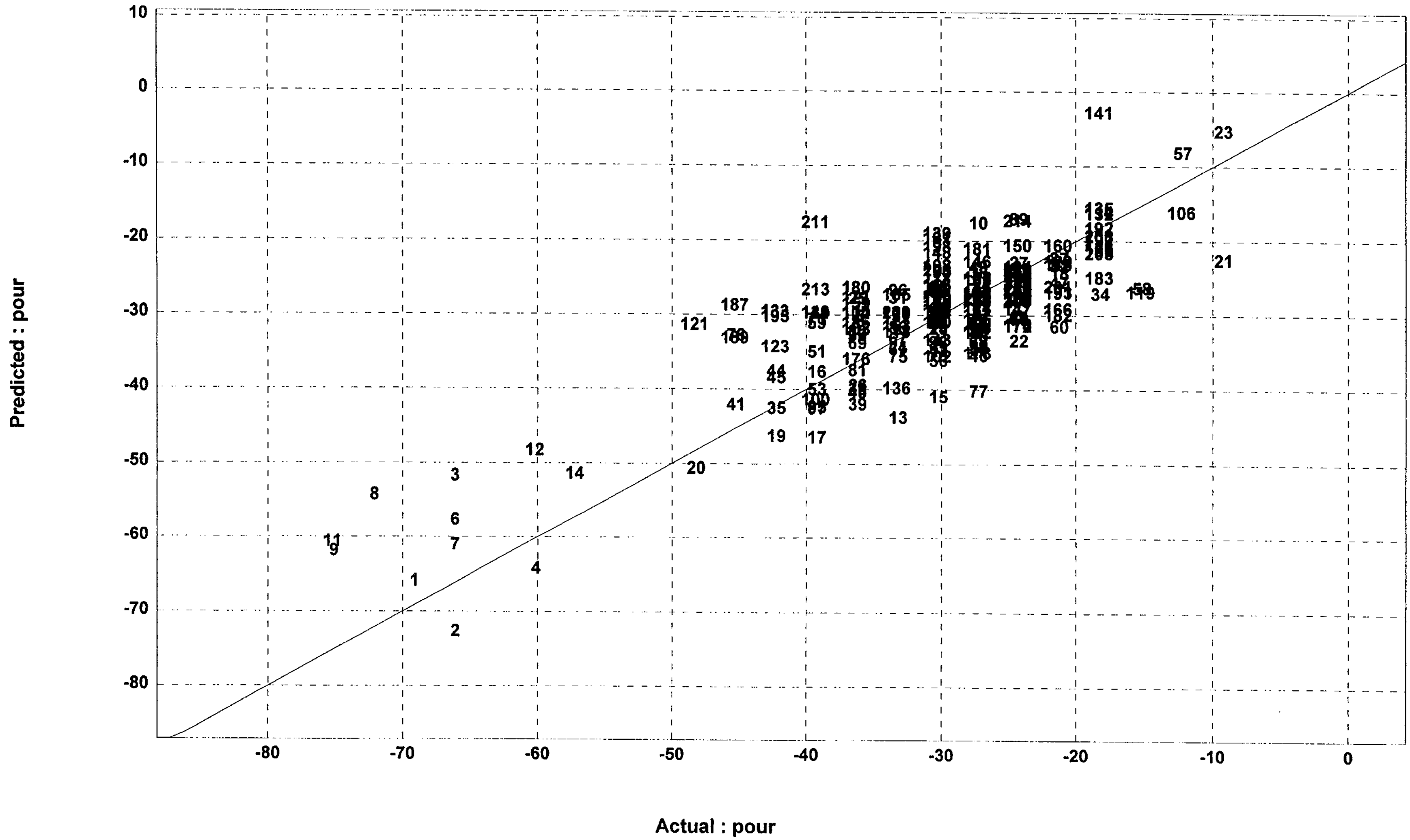
Total Factors: 11

Component: freeze

TE: 89.3892

R^2: .690697

RMSD: 6.11052



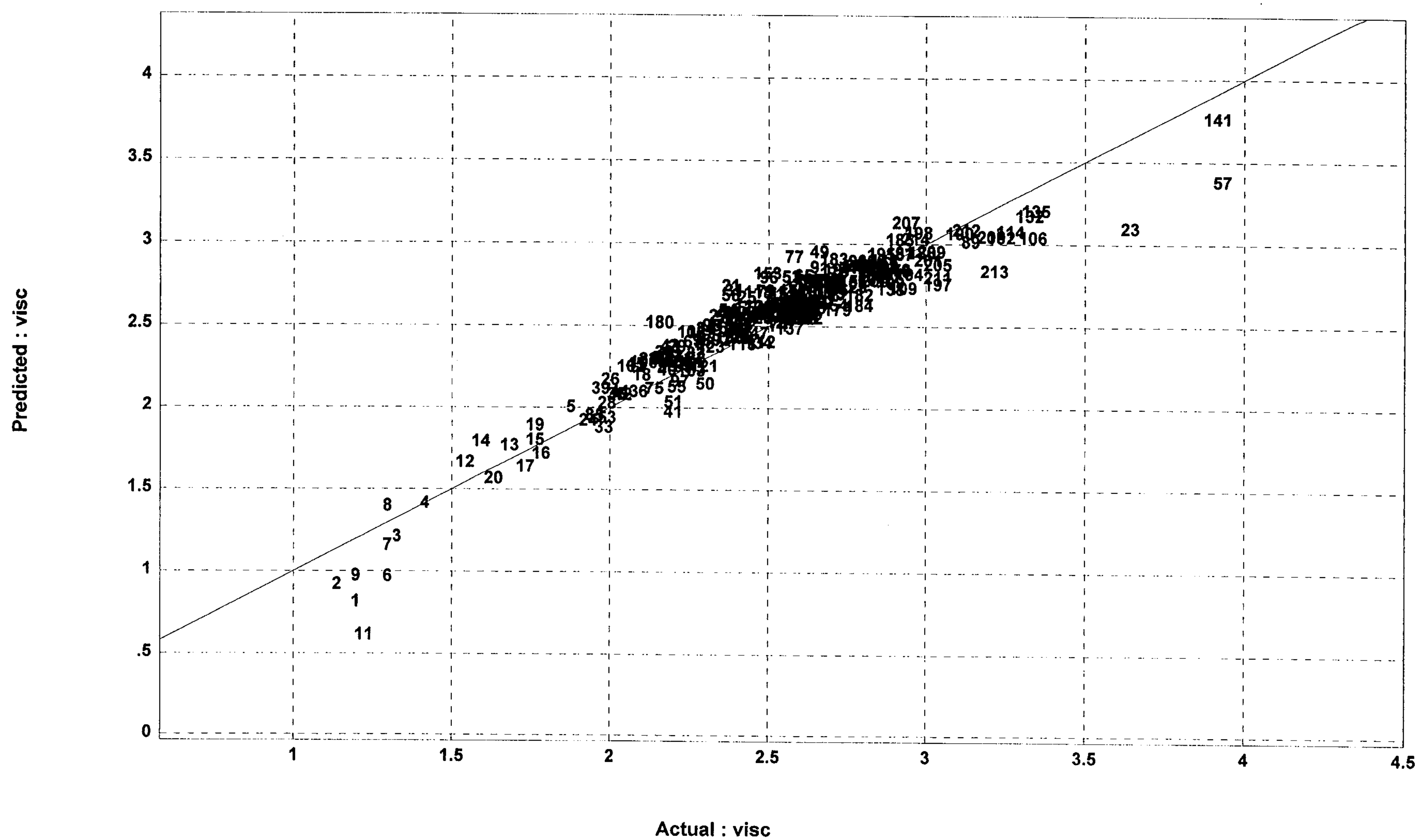
Total Factors: 11

Component: pour

TE: 2.18816

R^2: .892737

RMSD: .149579



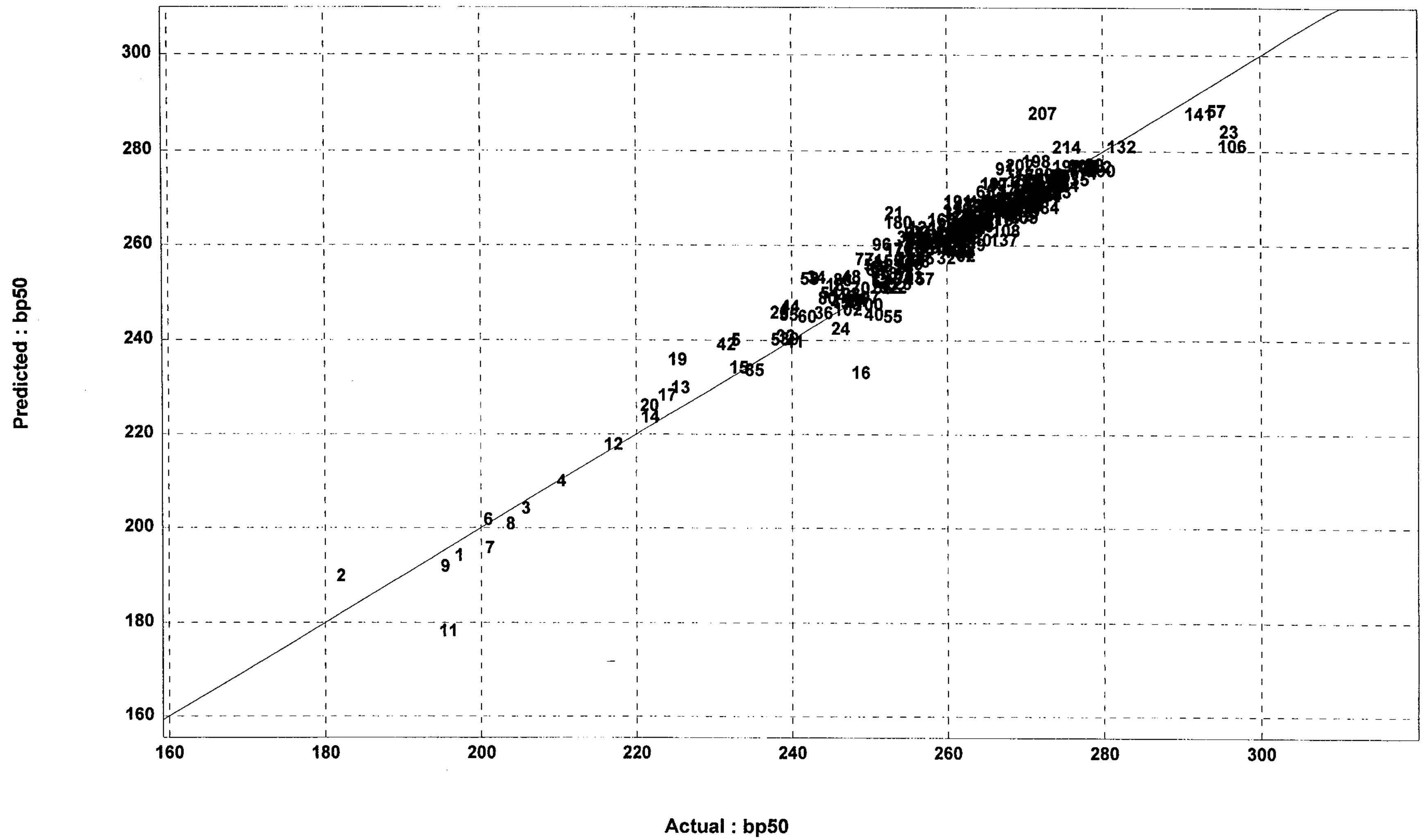
Total Factors: 19

Component: visc

TE: 63.5549

R^2: .940784

RMSD: 4.34453



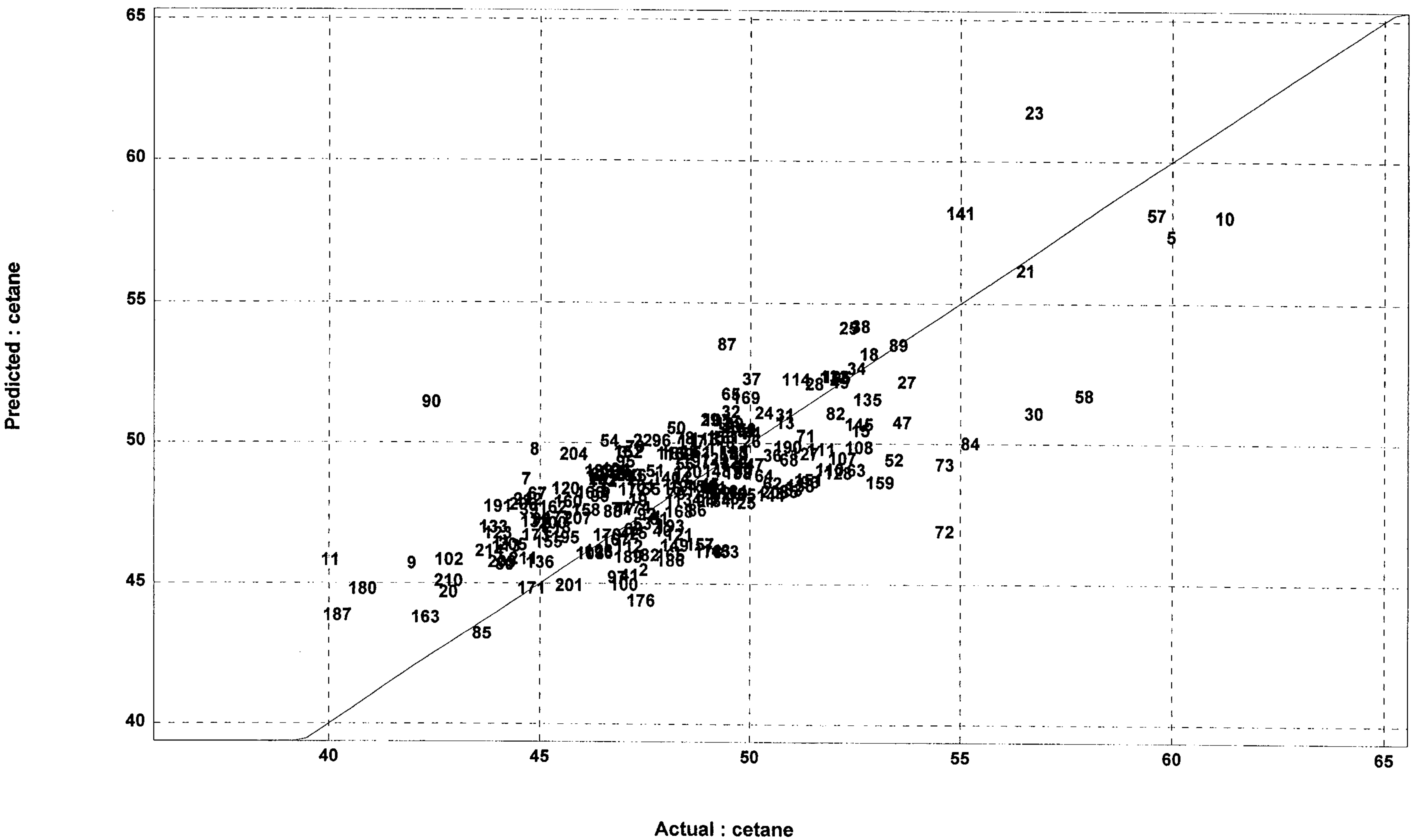
Total Factors: 17

Component: bp50

TE: 32.6002

R^2: .554611

RMSD: 2.2285



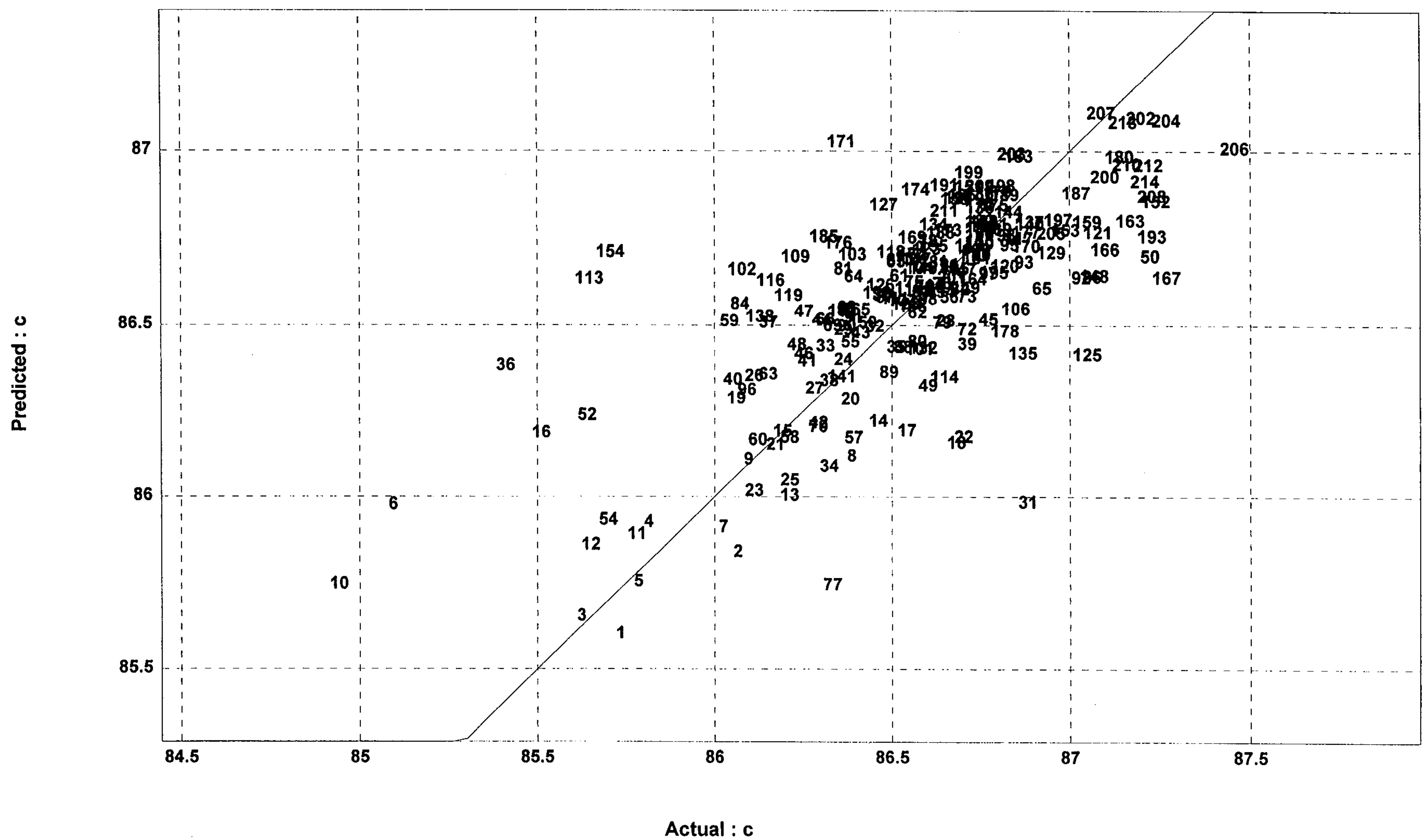
Total Factors: 7

Component: cetane

TE: 4.05491

R^2: .507183

RMSD: .277188



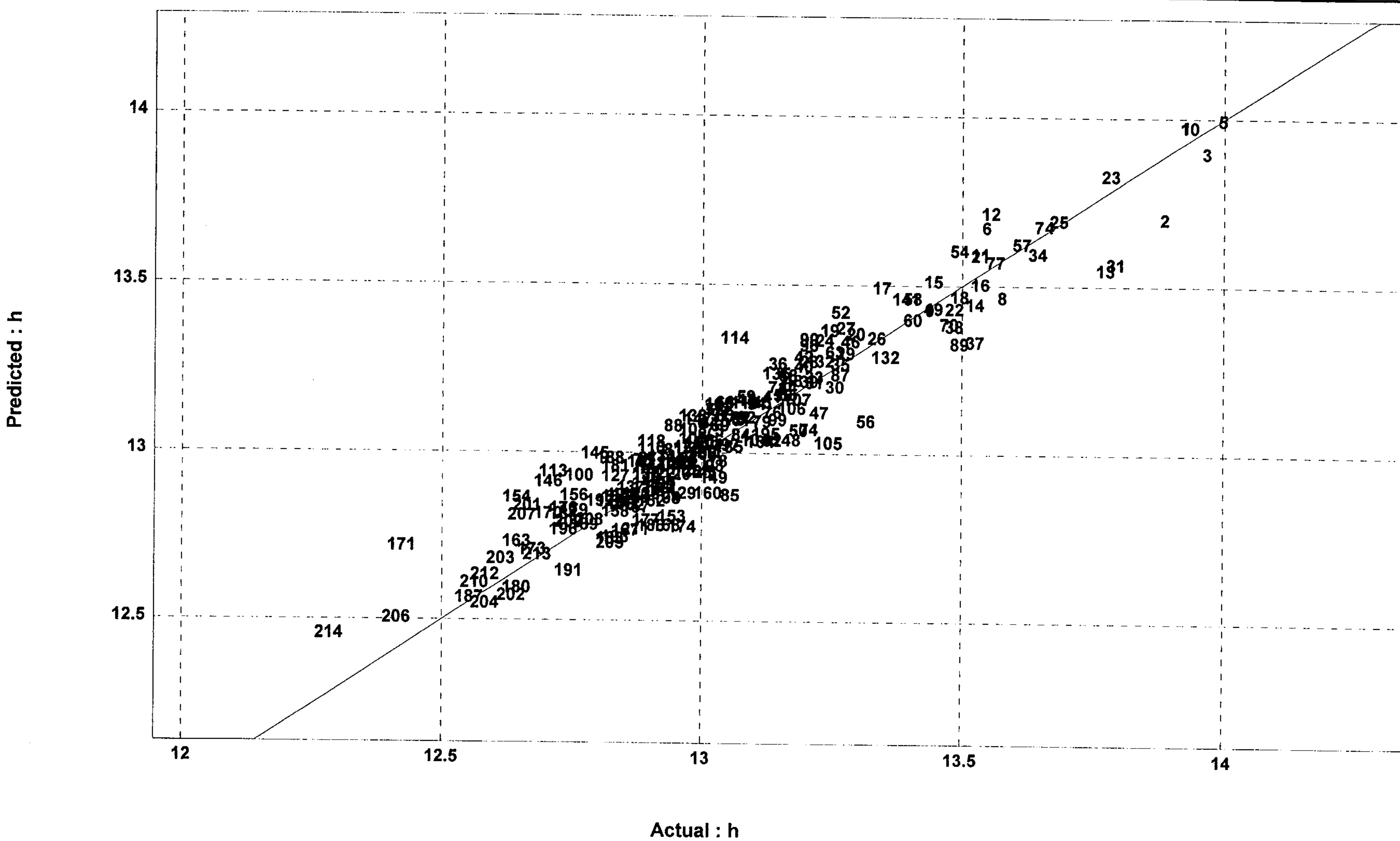
Total Factors: 4

Component: c

TE: 1.31454

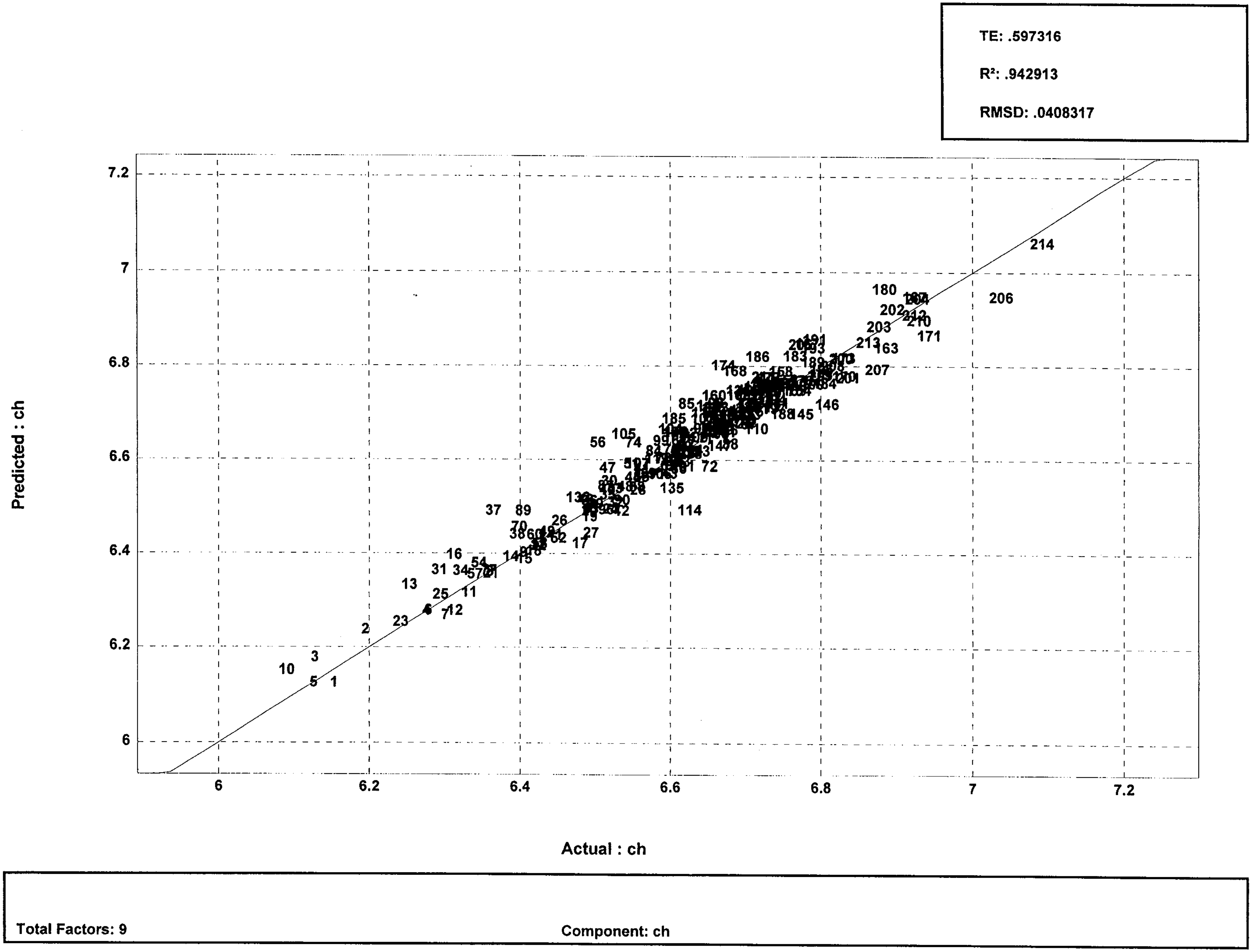
R^2: .911545

RMSD: .0898604

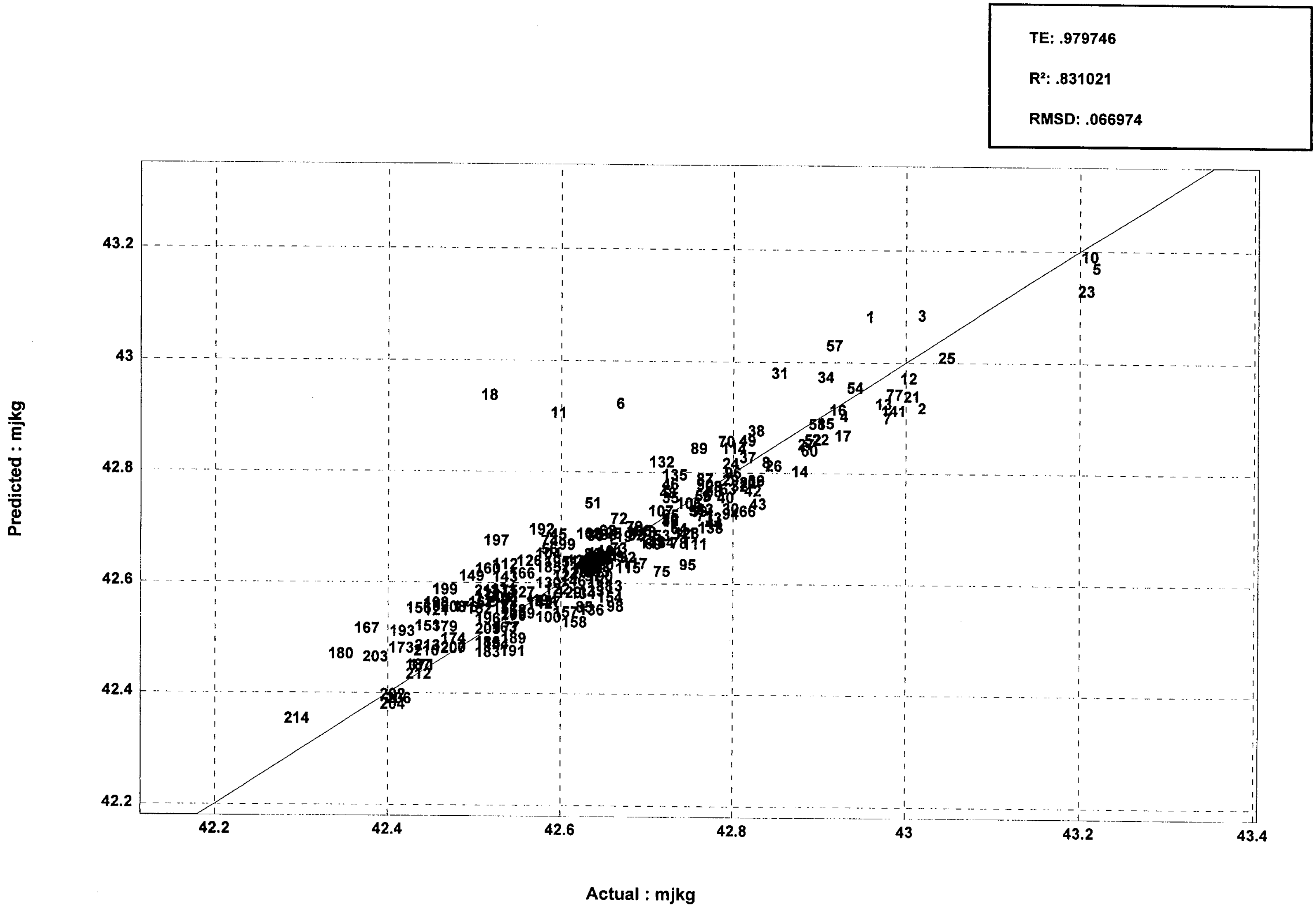


Total Factors: 8

Component: h



TE: .597316
R²: .942913
RMSD: .0408317
Predicted : ch
Actual : ch
7.2
7
6.8
6.6
6.4
6.2
6
Total Factors: 9
Component: ch



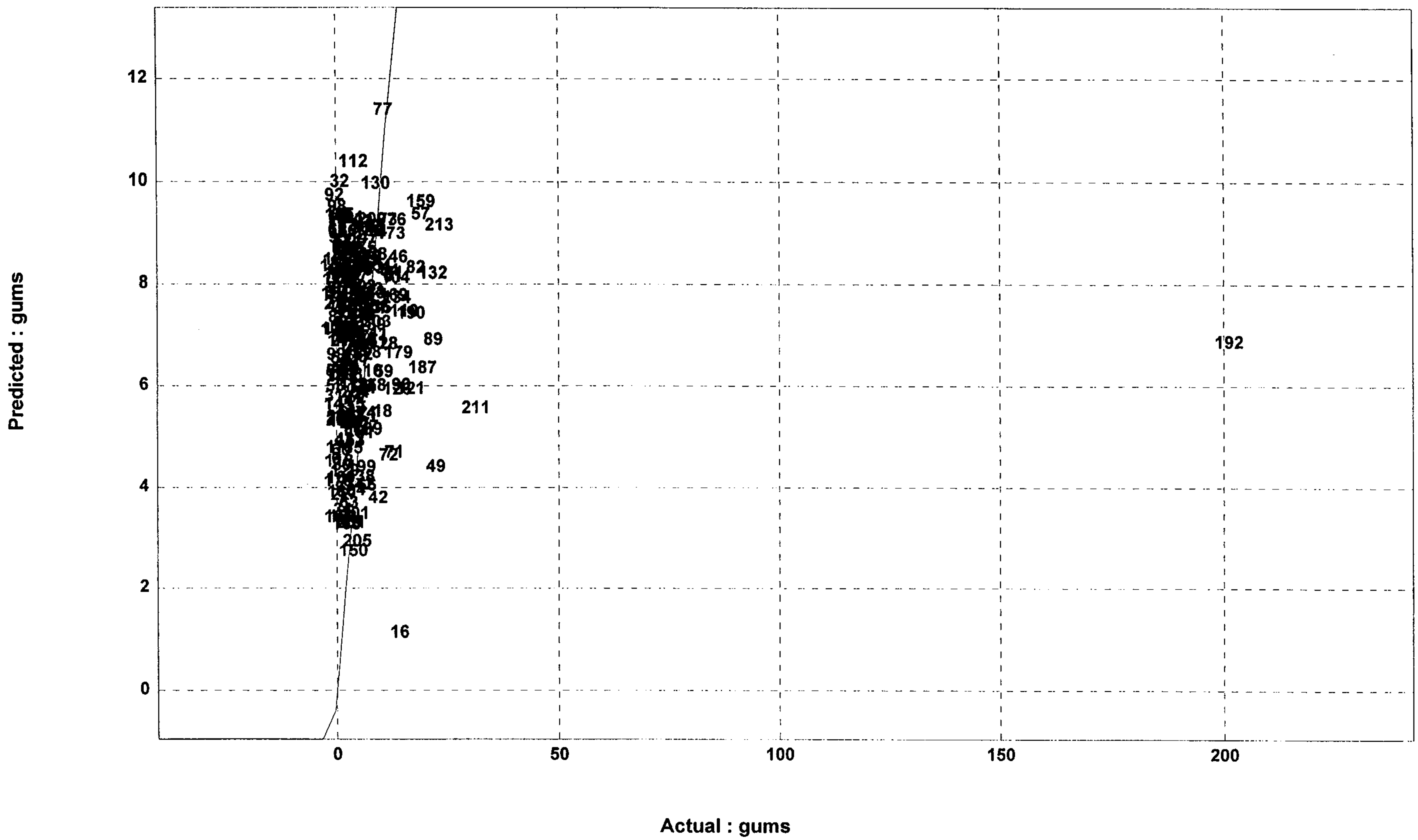
Total Factors: 7

Component: mjkg

TE: 211.641

R^2: .000755416

RMSD: 14.4675



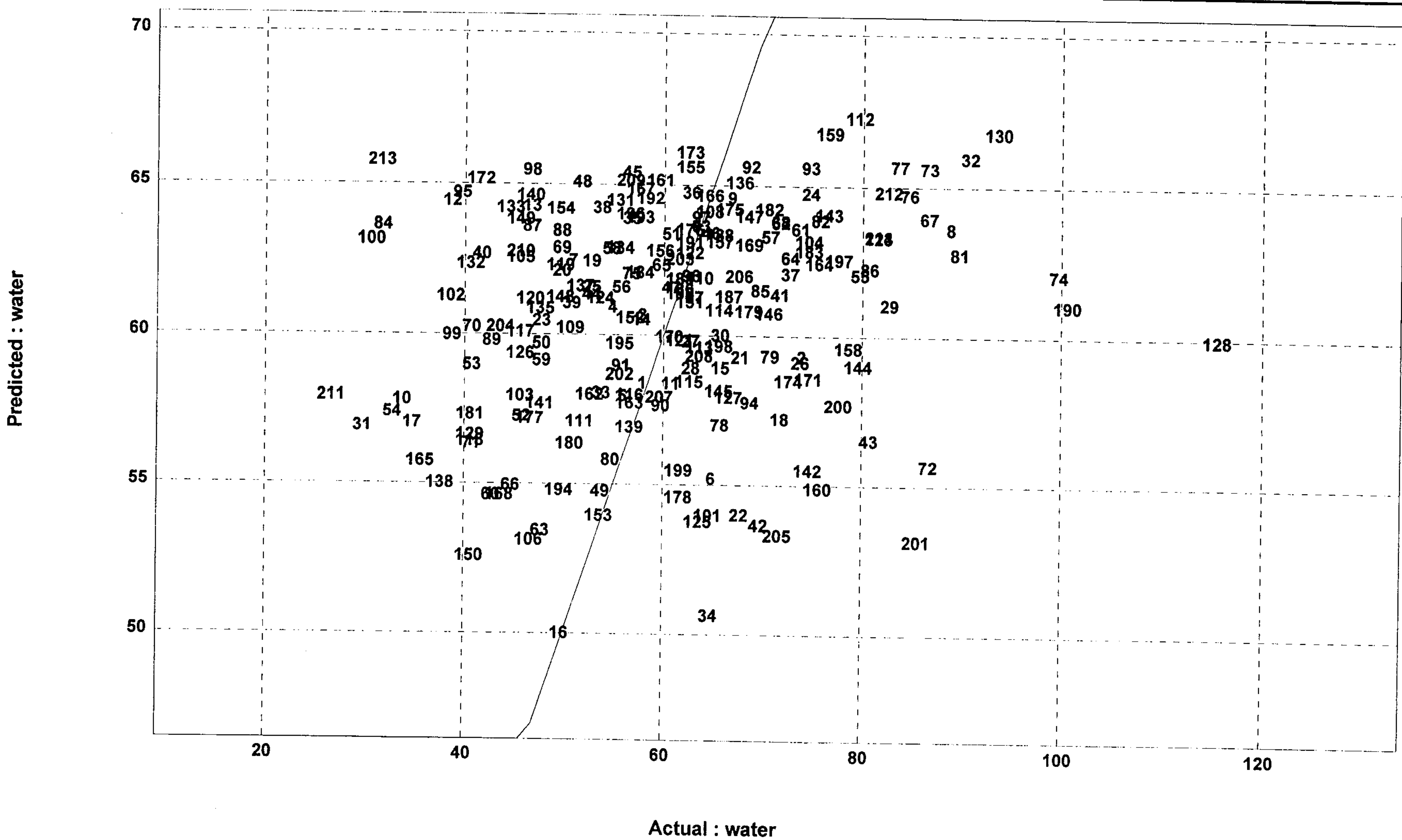
Total Factors: 1

Component: gums

TE: 210.016

R²: .038936

RMSD: 14.3564



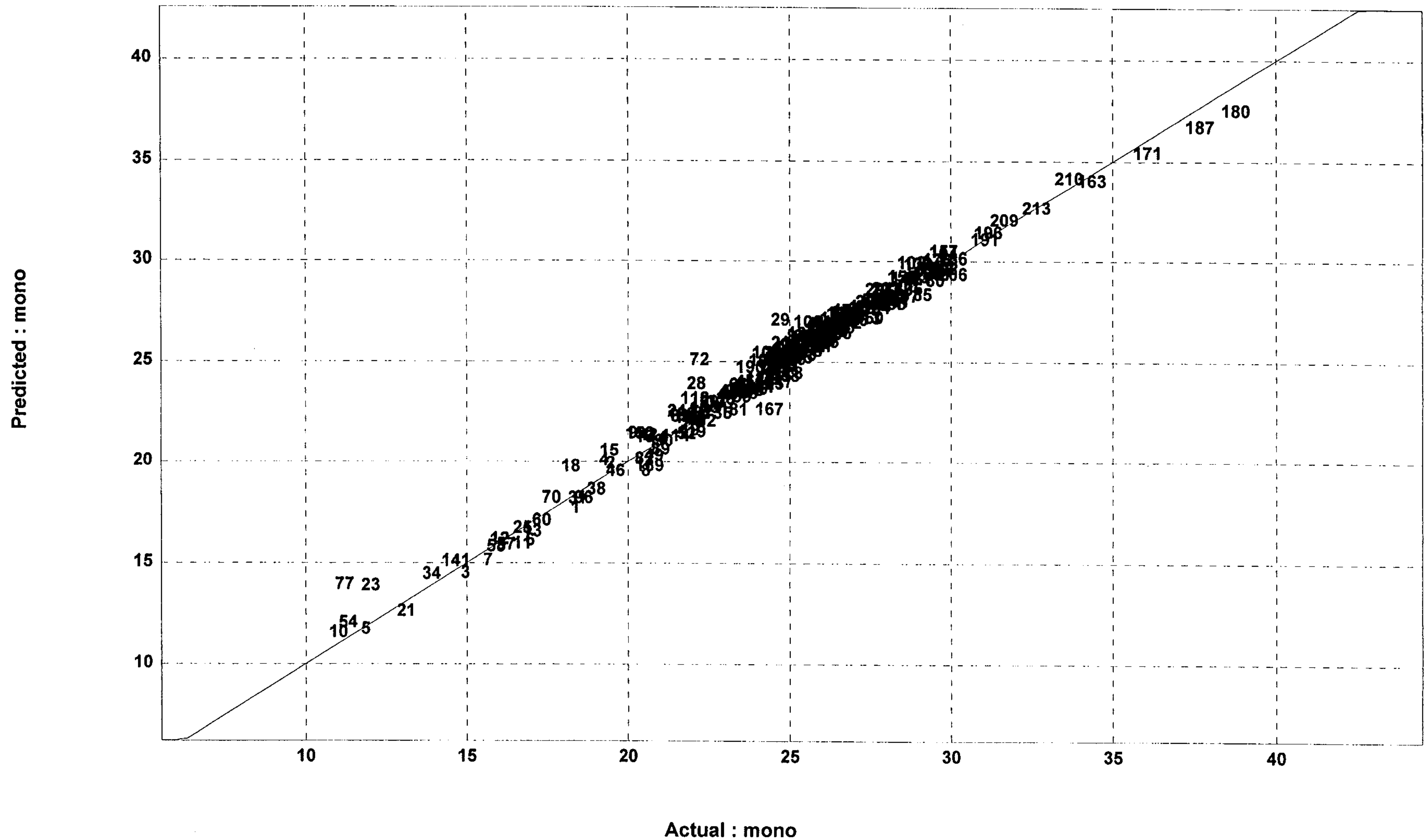
Total Factors: 1

Component: water

TE: 8.89012

R^2: .981562

RMSD: .607716



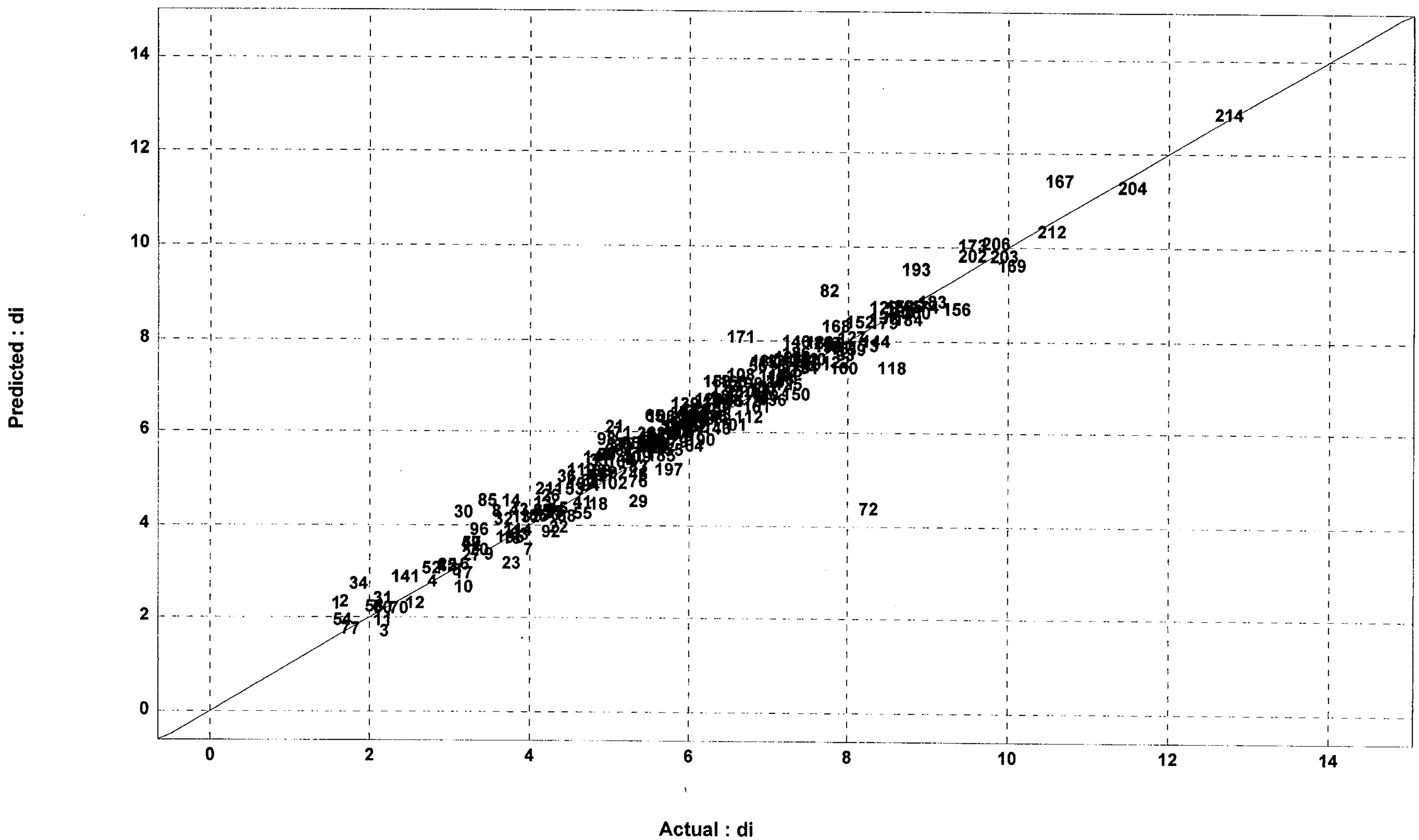
Total Factors: 17

Component: mono

TE: 6.80885

R^2: .946153

RMSD: .465443



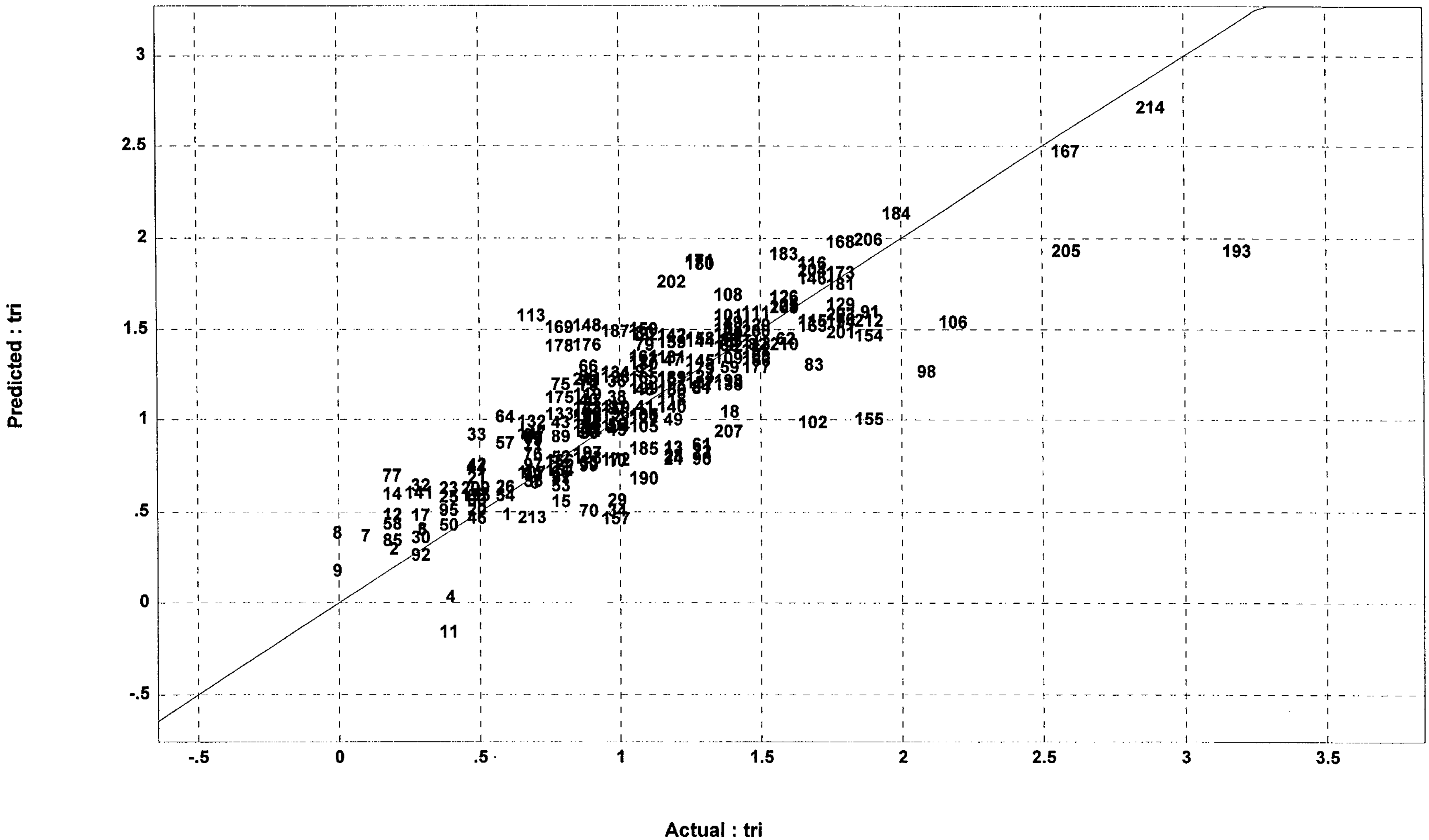
Total Factors: 16

Component: di

TE: 4.18411

R²: .686489

RMSD: .28602



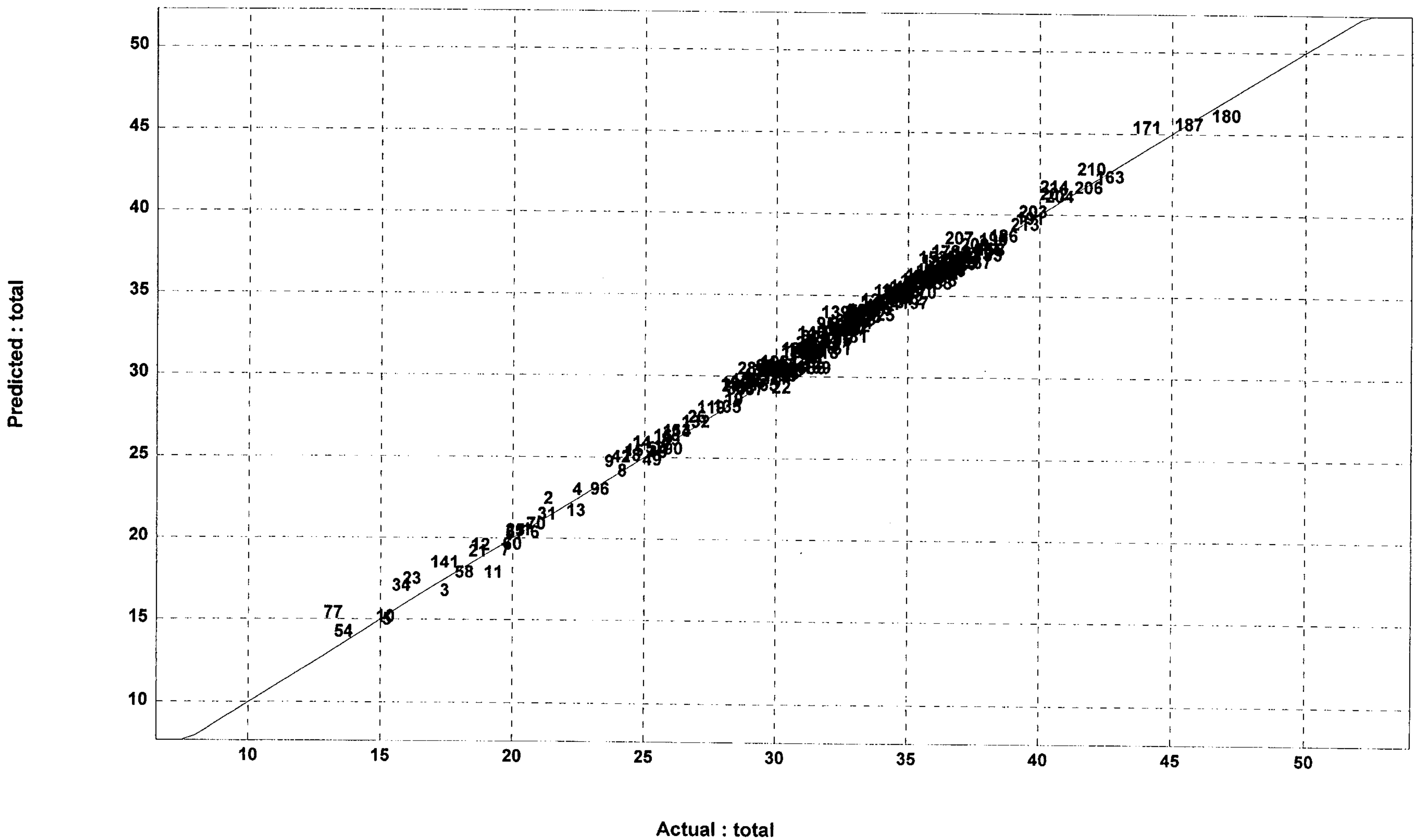
Total Factors: 13

Component: tri

TE: 7.98953

R^2: .991779

RMSD: .546153



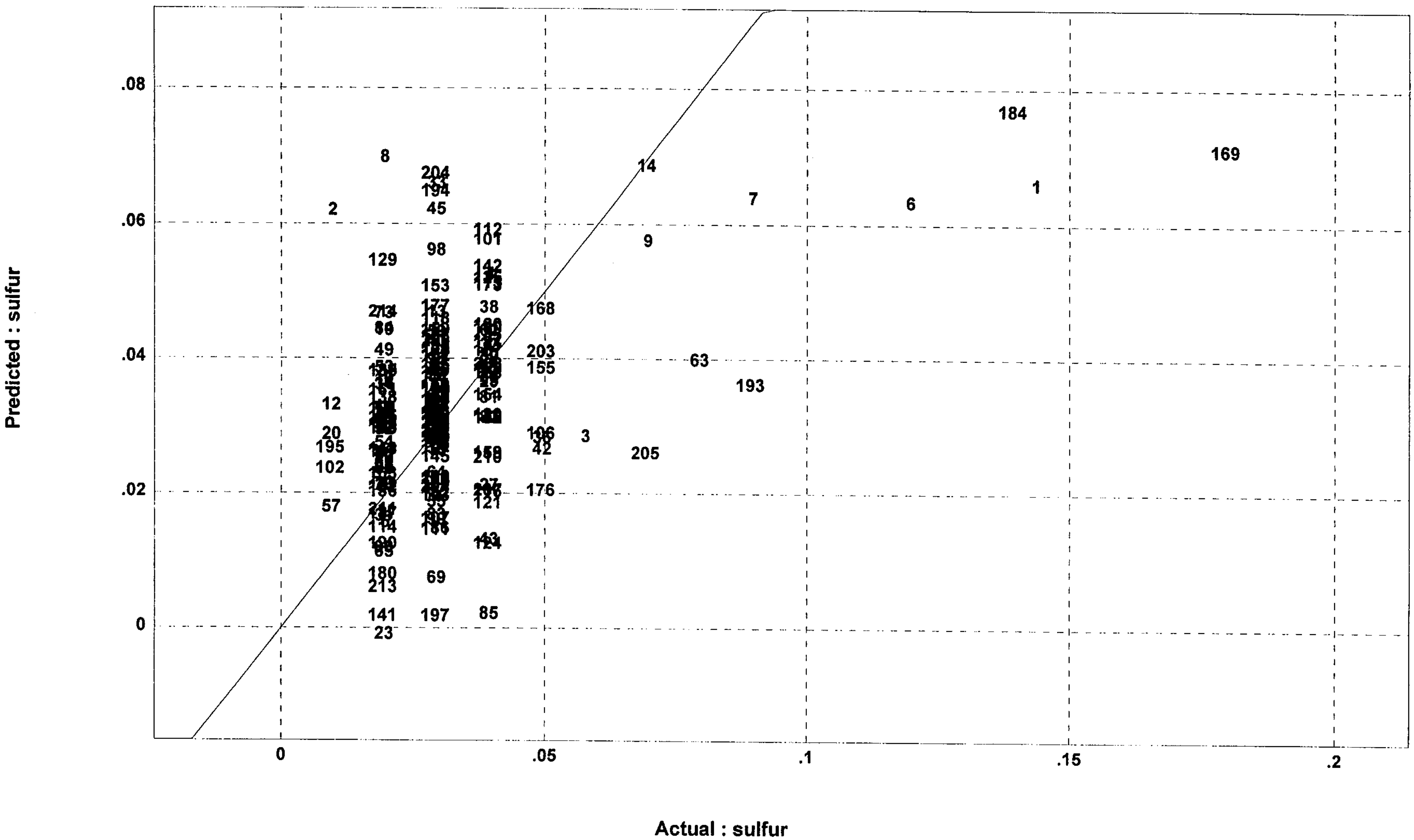
Total Factors: 19

Component: total

TE: .268965

R^2: .199239

RMSD: .018386



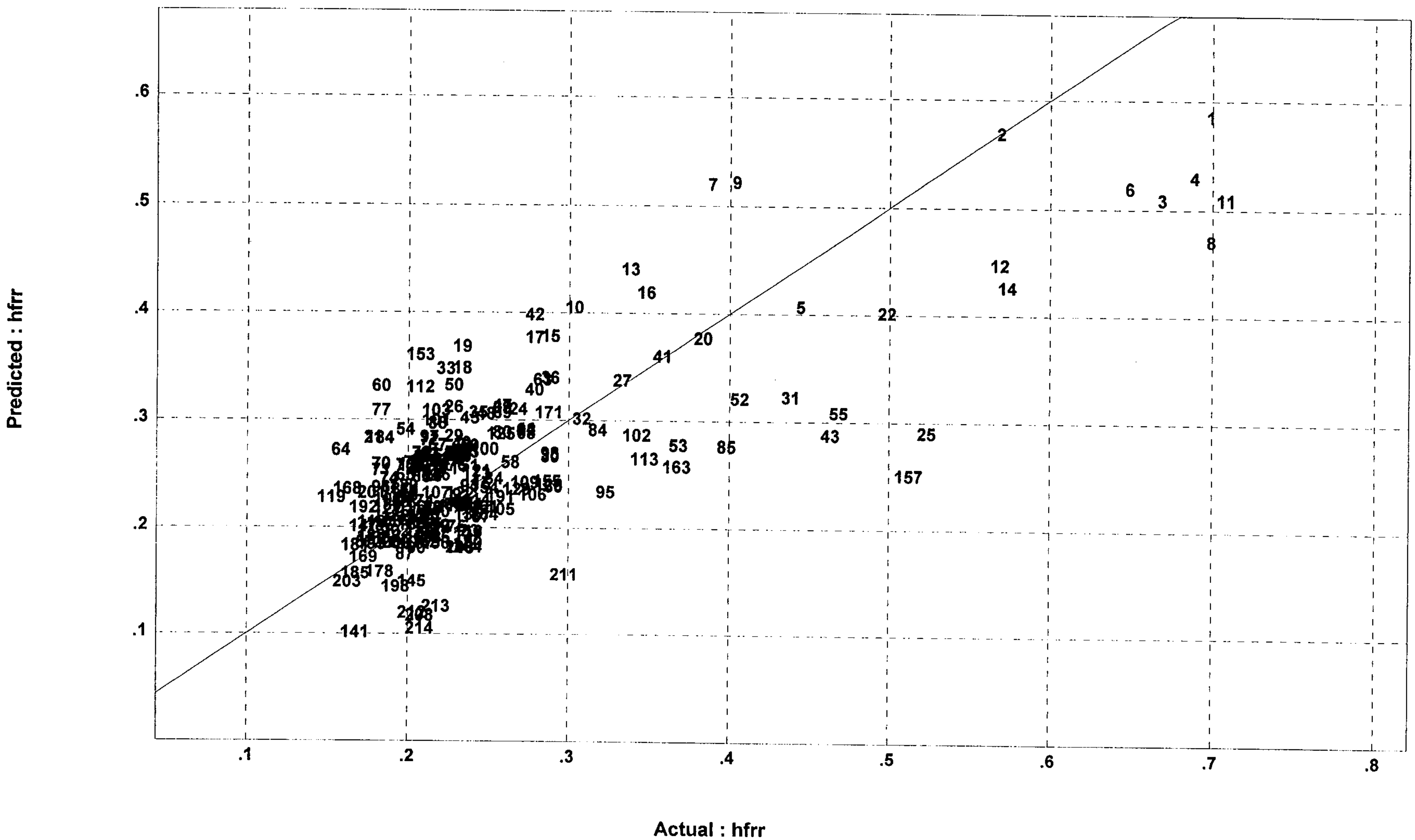
Total Factors: 14

Component: sulfur

TE: 1.01512

R^2: .561464

RMSD: .0693925



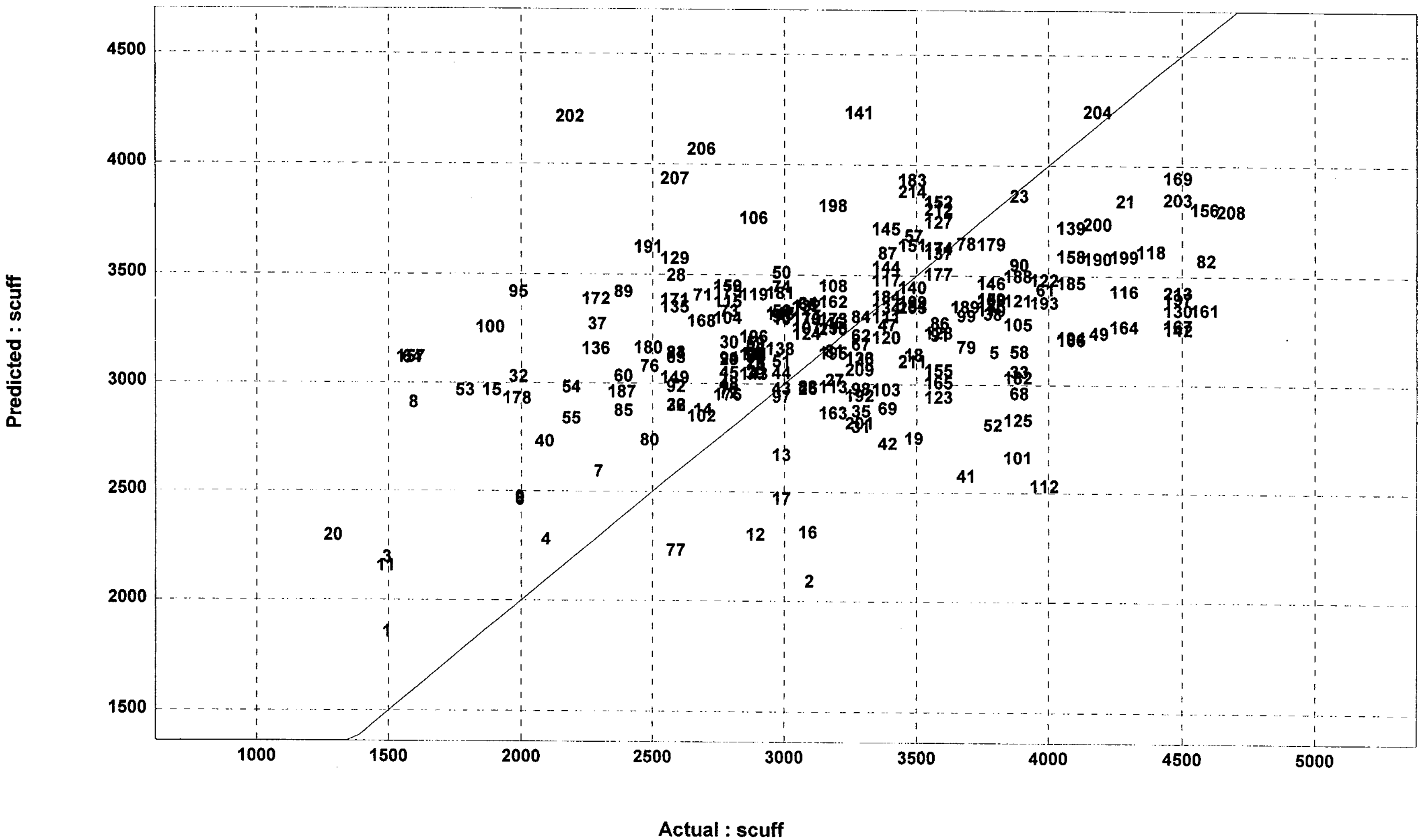
Total Factors: 8

Component: hfrr

TE: 9240.97

R^2: .202429

RMSD: 631.7



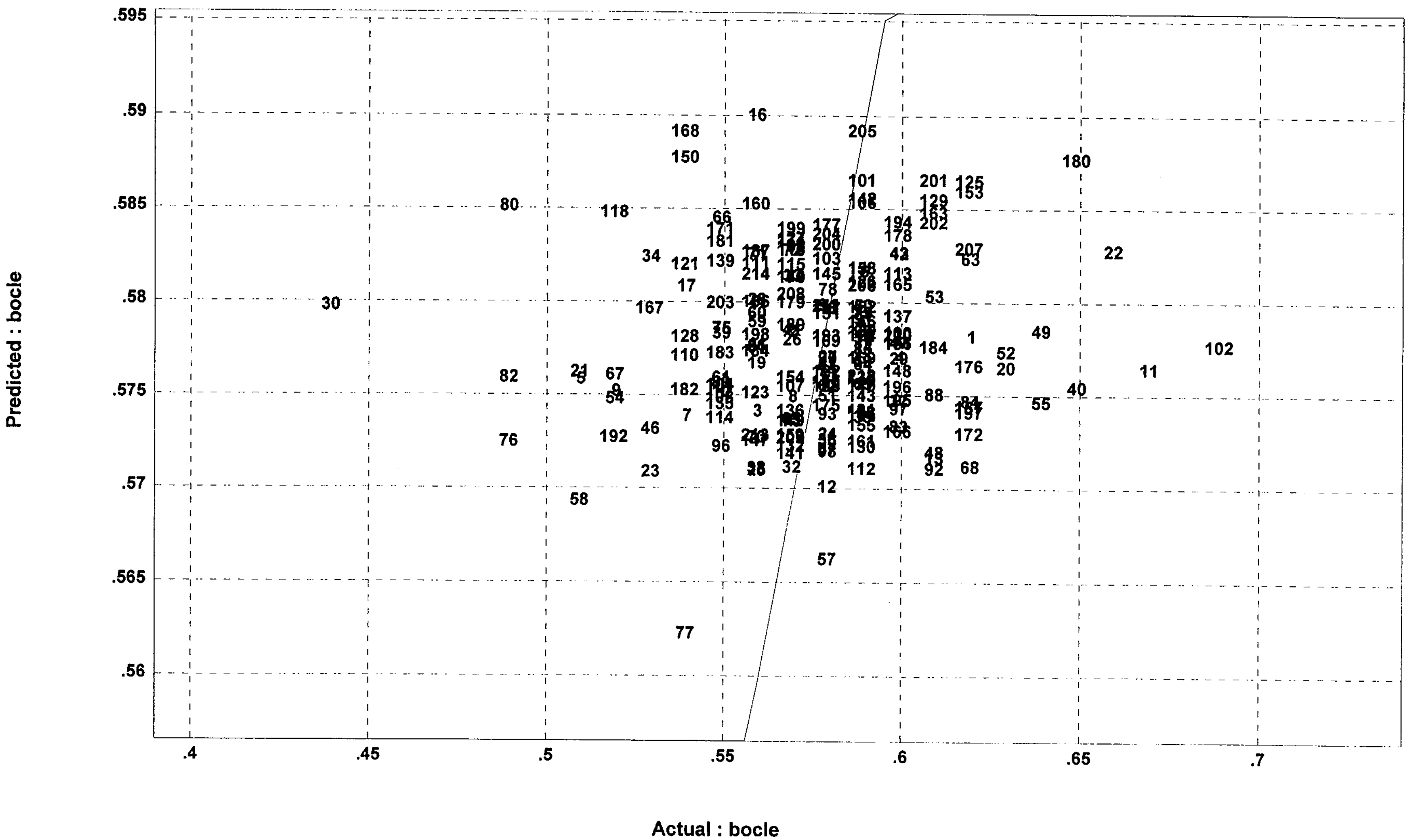
Total Factors: 6

Component: scuff

TE: .46294

R^2: .00363924

RMSD: .0316459

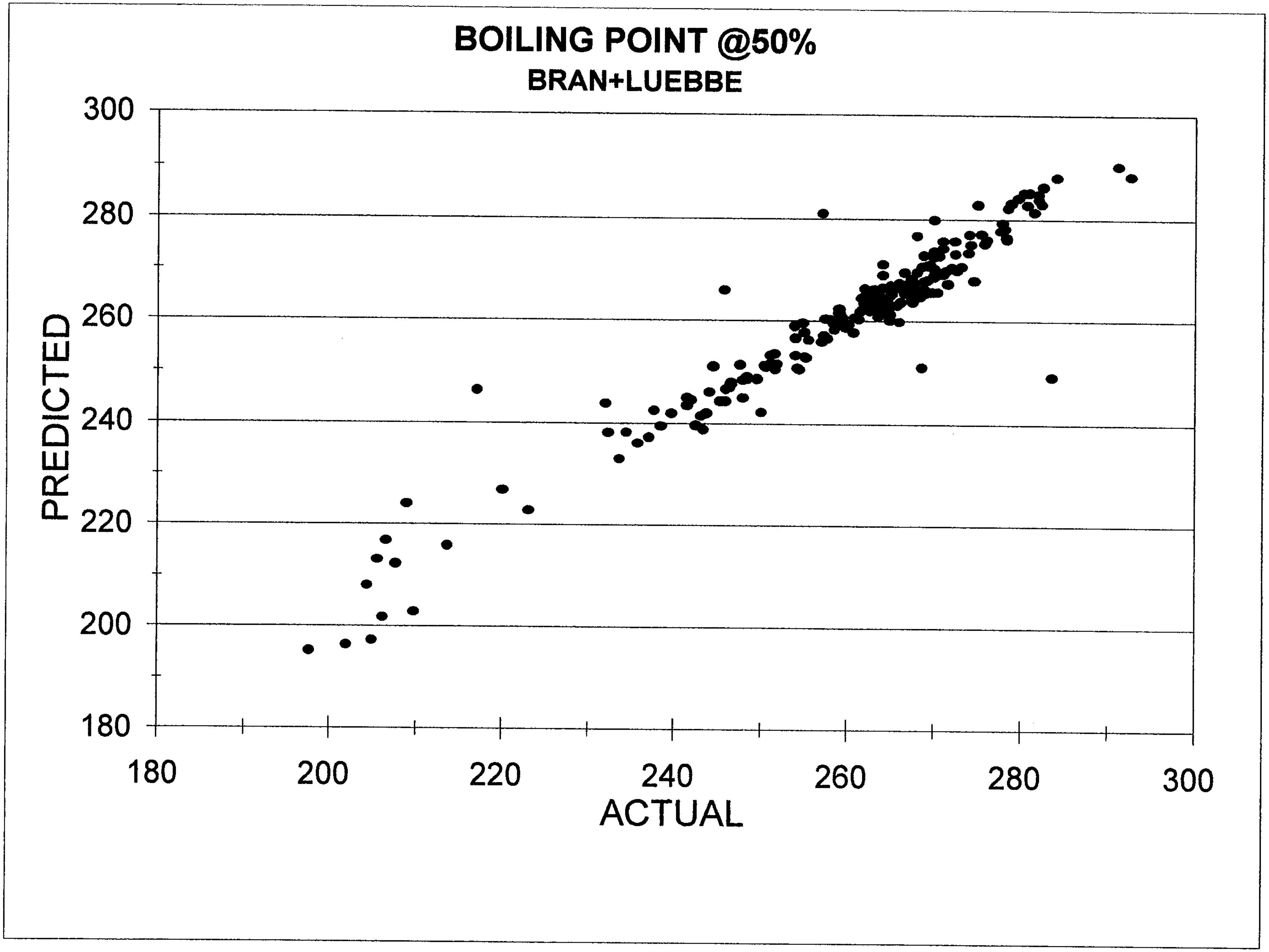


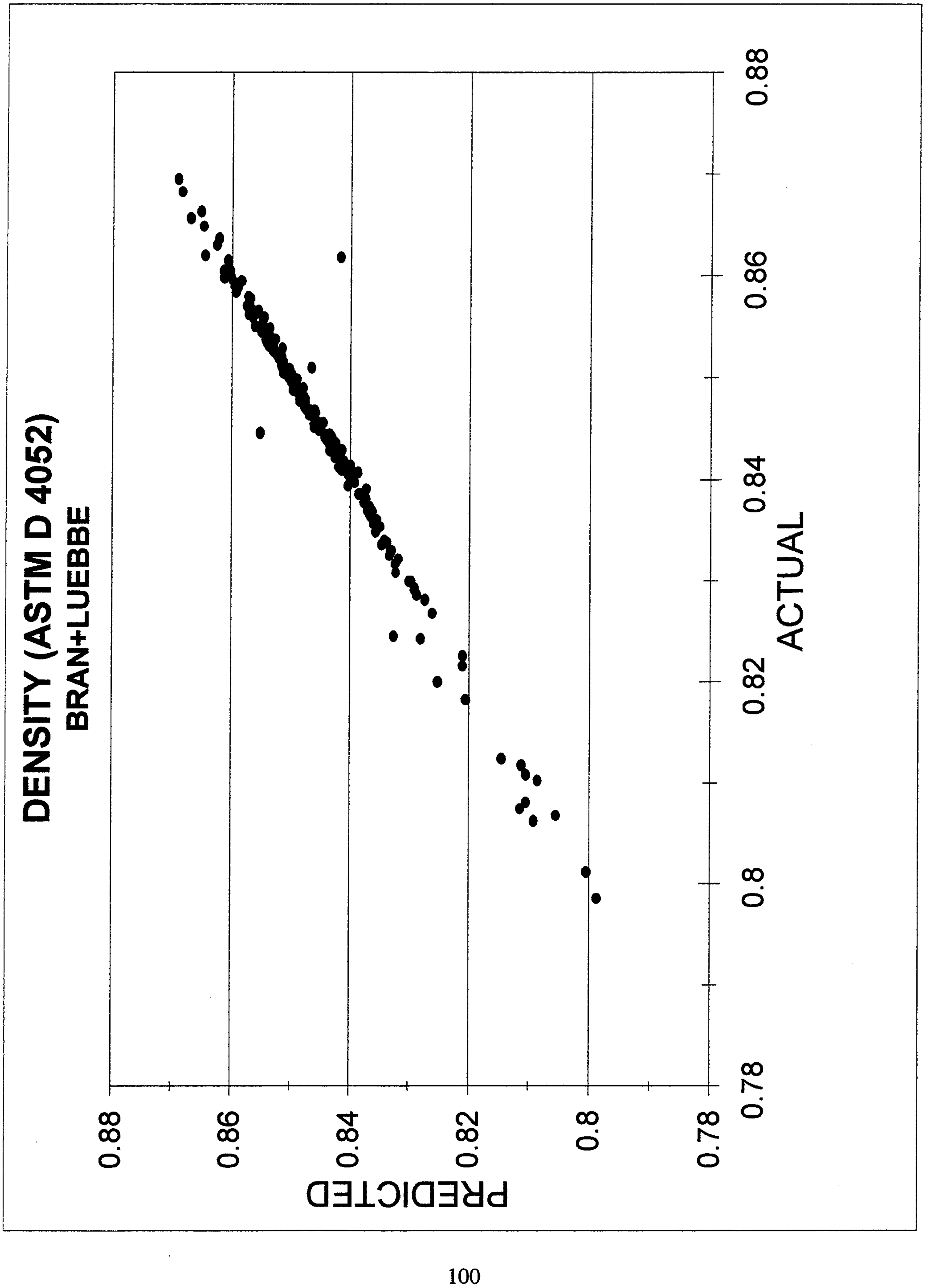
Total Factors: 1

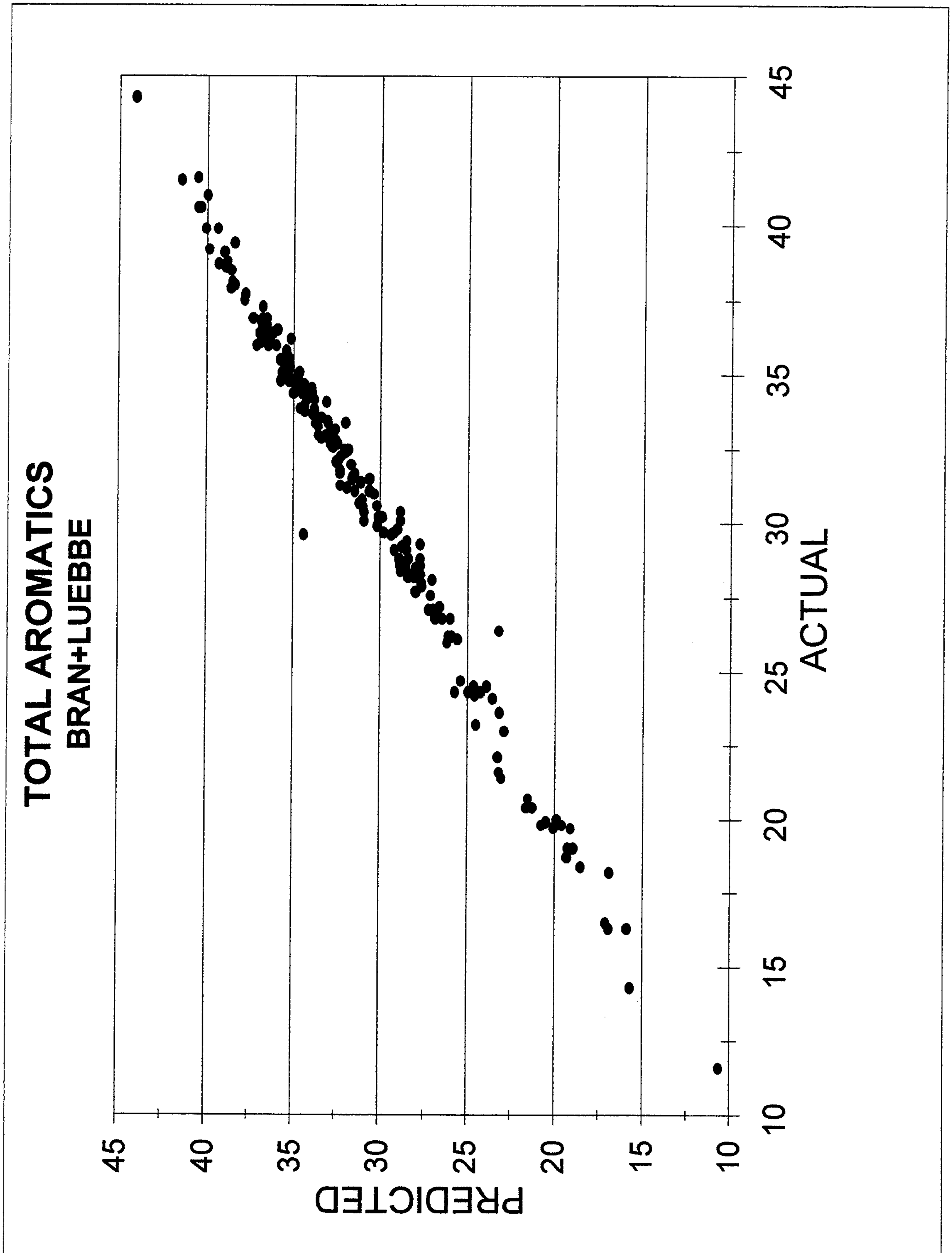
Component: bocle

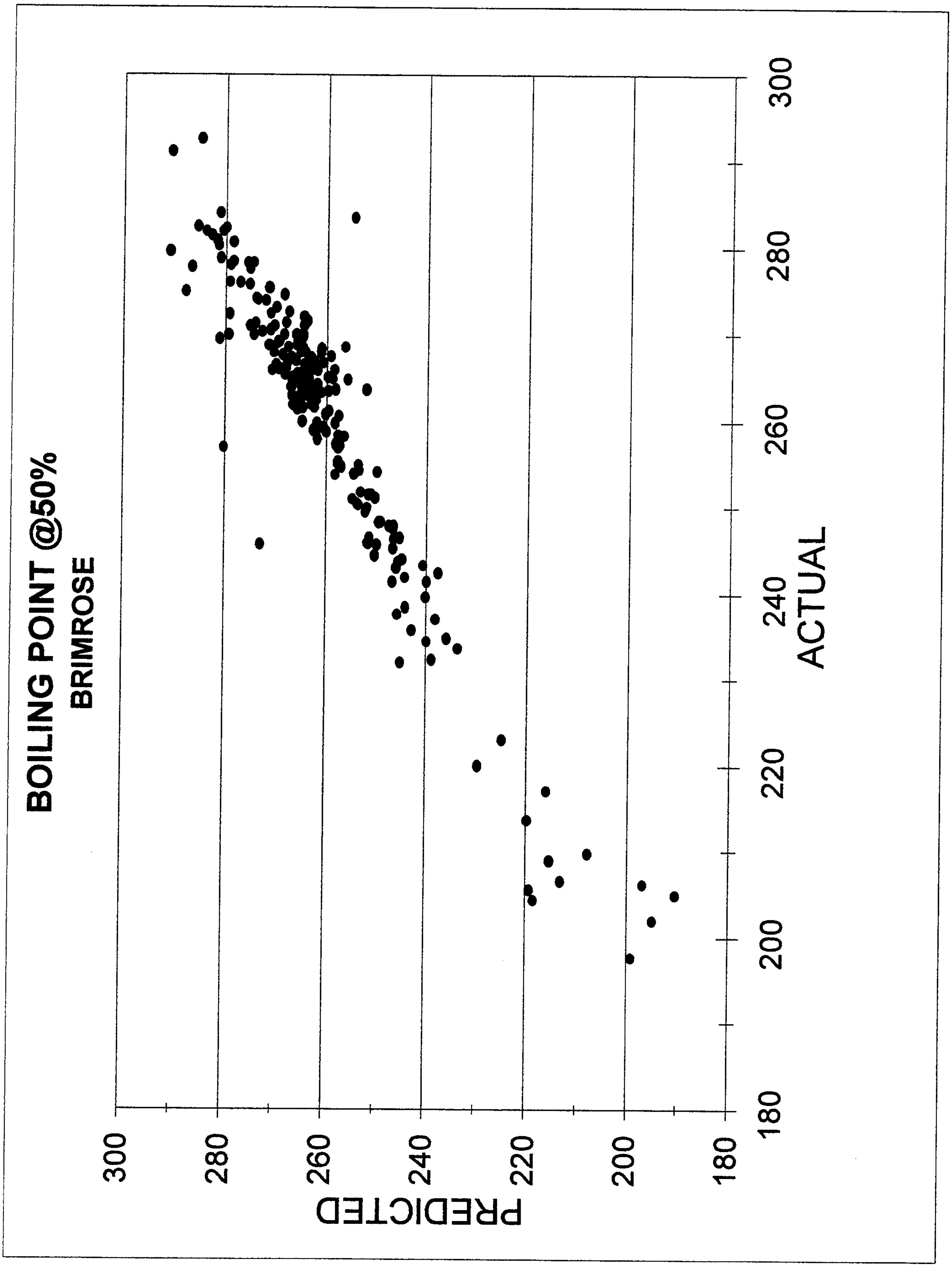
APPENDIX B

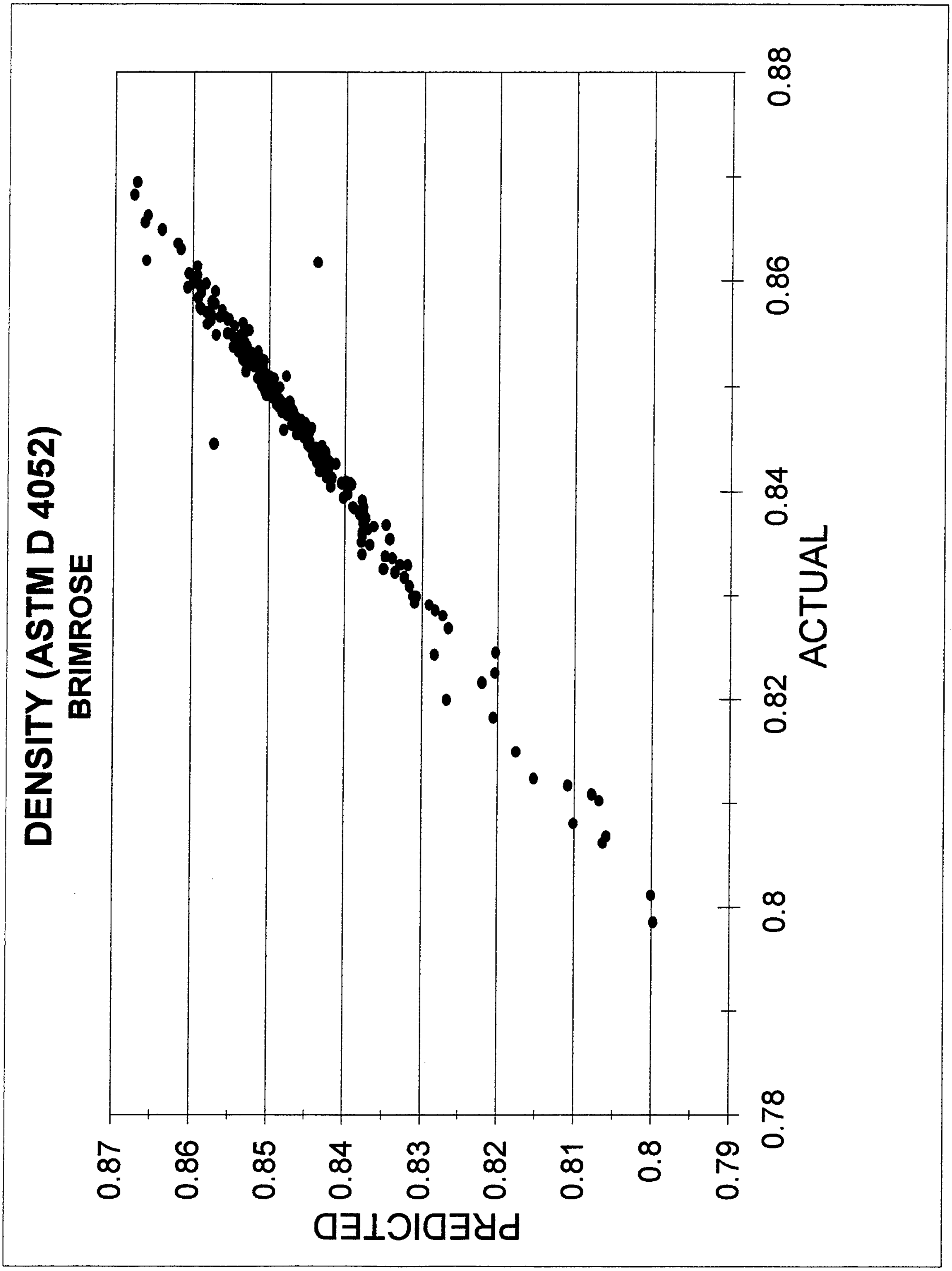
Results of Analyses

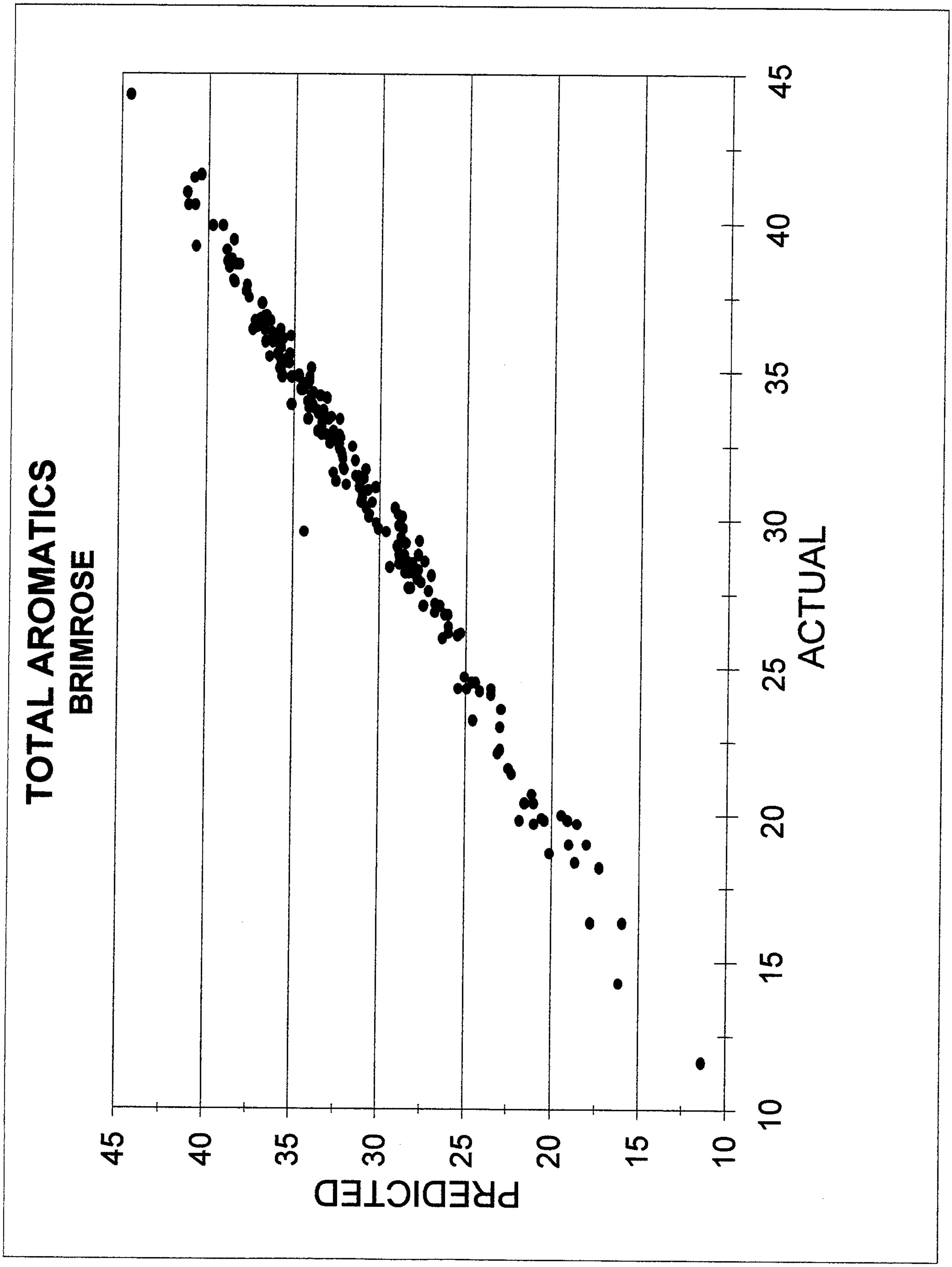

BOILING POINT @50%
BRAN+LUEBBE
PREDICTED
ACTUAL
180
200
220
240
260
280
300

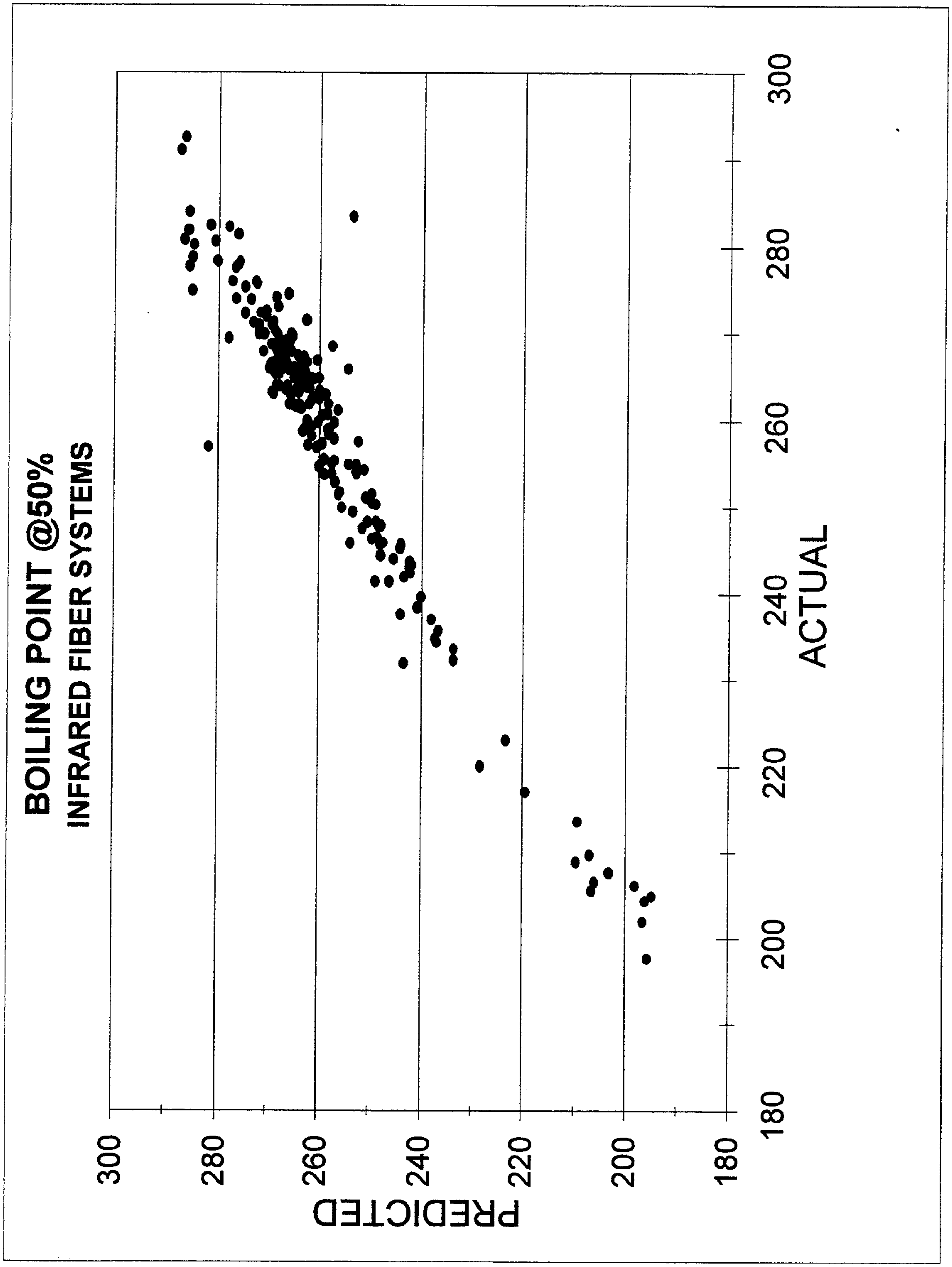

DENSITY (ASTM D 4052)
BRAN+LUEBBE
PREDICTED
ACTUAL
0.78
0.8
0.82
0.84
0.86
0.88


TOTAL AROMATICS
BRAN+LUEBBE
PREDICTED
ACTUAL
10
15
20
25
30
35
40
45

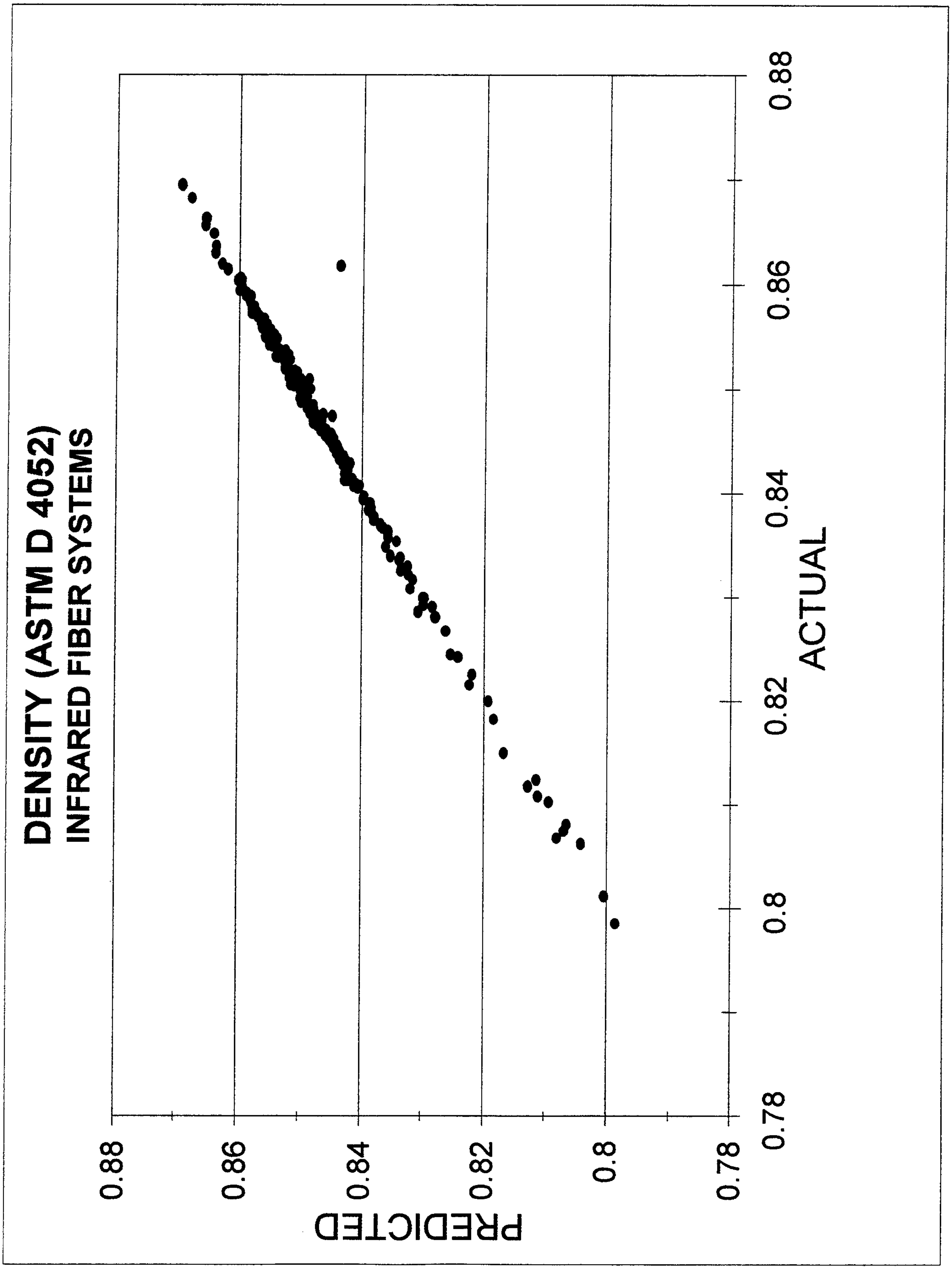

BOILING POINT @50%
BRIMROSE
PREDICTED
ACTUAL
180
200
220
240
260
280
300

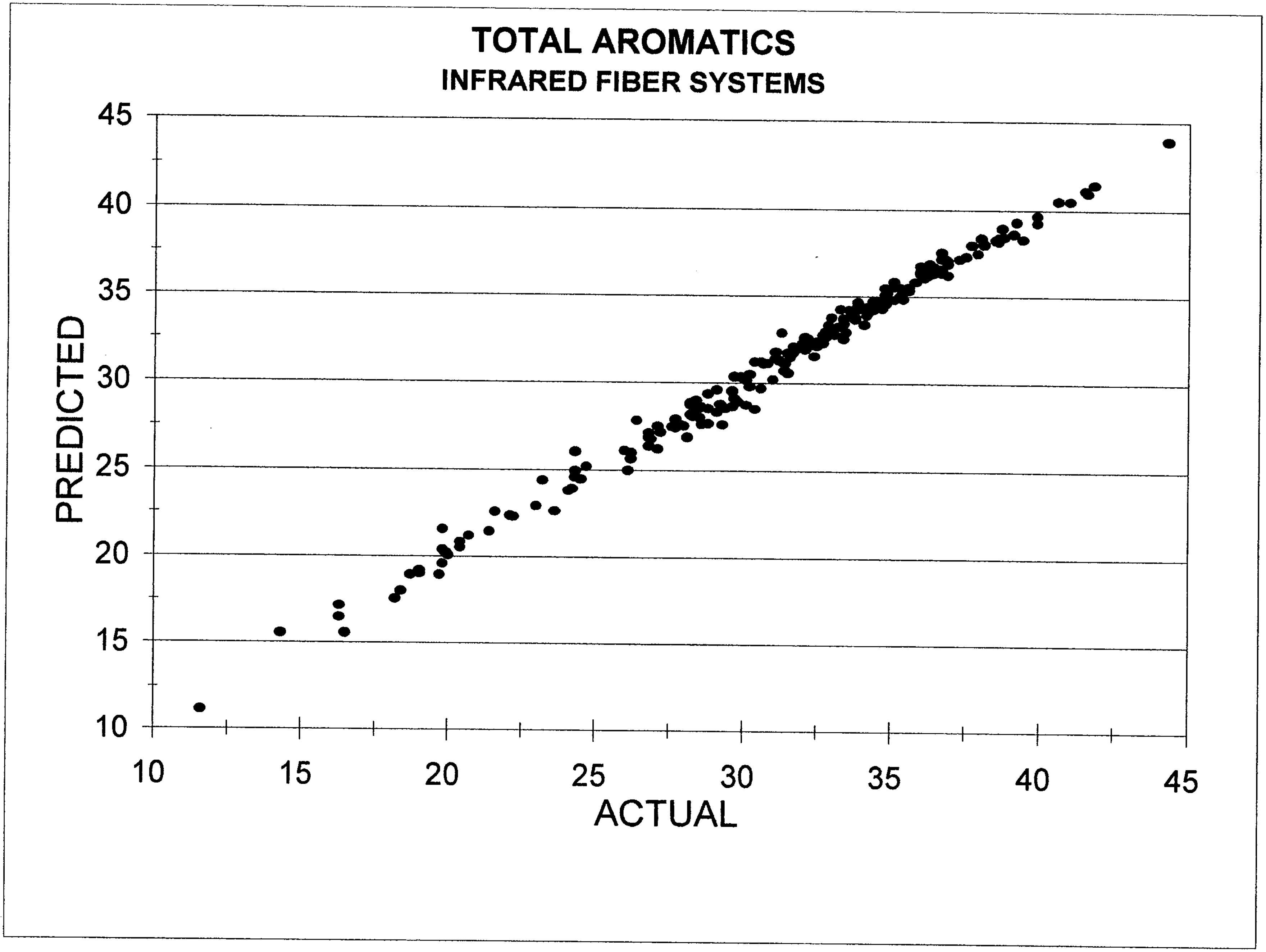

DENSITY (ASTM D 4052)
BRIMROSE
PREDICTED
ACTUAL
0.87
0.86
0.85
0.84
0.83
0.82
0.81
0.8
0.79
0.78
0.8
0.82
0.84
0.86
0.88


TOTAL AROMATICS
BRIMROSE
PREDICTED
ACTUAL
10
15
20
25
30
35
40
45



BOILING POINT @50%
INFRARED FIBER SYSTEMS
PREDICTED
300
280
260
240
220
200
180
ACTUAL
180
200
220
240
260
280
300


DENSITY (ASTM D 4052)
INFRARED FIBER SYSTEMS
PREDICTED
0.78
0.8
0.82
0.84
0.86
0.88
ACTUAL
0.78
0.8
0.82
0.84
0.86
0.88


TOTAL AROMATICS
INFRARED FIBER SYSTEMS
PREDICTED
ACTUAL
10
15
20
25
30
35
40
45

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